Quarterly Progress Report
Determination of [redacted] Emissivity of Materials
Report No. PWA-2128

Report Period: July 1 through September 30, 1962

Contract NASw-104 with 8 Amendments

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ERRATA

Please make the following corrections to the "Quarterly Progress Report, Determination of Emissivity of Materials," Report No. PWA-2128:

1. Page 30, delete the word "heating" where it appears in the legend.

2. Page 63, change "200°F" to "2000°F" in the legend.

UNITED AIRCRAFT CORPORATION
Pratt & Whitney Aircraft

FOREWORD

This report describes the research activity carried out in fulfillment of Contract NASw-104 as modified by Amendments 1 through 8, during the period from July 1 through September 30, 1962.

ABSTRACT

During the three month period covered by this report, work was continued in support of NASA space power systems. An aluminum phosphate-bonded mixture of nickel-chrome spinel and silicon dioxide completed 8650 hours of endurance testing on a SNAP-8 finned-tube radiator segment. Flame-sprayed coatings of titania on SNAP-8 and Sunflower I sections completed 7810 hours and 7760 hours respectively. A fourth rig containing a SNAP-8 section with an aluminum phosphate-bonded mixture of silicon carbide and silicon dioxide completed 6530 hours of testing.

Emittance measurements were made on ten materials and on the substrate material, columbium-1 per cent zirconium alloy. The ten materials tested included zirconium silicate, magnesium aluminate, chromic oxide, ceric oxide, titania, barium titanate, calcium titanate, strontium titanate, iron-titanum-aluminum oxide, and iron-titanium oxide. Measurement of both total hemispherical and spectral normal emittances were made for all materials except ceric oxide and iron titanium oxide. Two different bonding methods were used for chromic oxide and calcium titanate and three different methods were used for zirconium silicate. One zirconium silicate coating was bonded to type 310 stainless steel rather than columbium-1 per cent zirconium to determine the influence on coating emittance of the type substrate used. Specimens of zirconium silicate and iron-titanium oxide were endurance tested.

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I COATING ENDURANCE TESTS IN SUPPORT OF NASA SPACE POWER SYSTEMS

Work was continued during this report period in support of the SNAP-8 and Sunflower I space power systems. Endurance tests, each scheduled to run for ten thousand hours, continued on four finned-tube radiator segment endurance rigs. A description of the work on each rig follows.

A. <u>Rig No. 1, SNAP-8 Test Section</u>

A mixture of nickel-chrome spinel ($NiO \cdot Cr_2O_3$) and silicon dioxide are aluminum phosphate-bonded onto this test section. Report PWA-1994, page 12, describes the various tests this material has been subjected to. At the end of September accumulated test time totaled 8650 hours.

B. <u>Rig No. 2, SNAP-8 Test Section</u>
<u>Rig No. 3, Sunflower I Test Section</u>

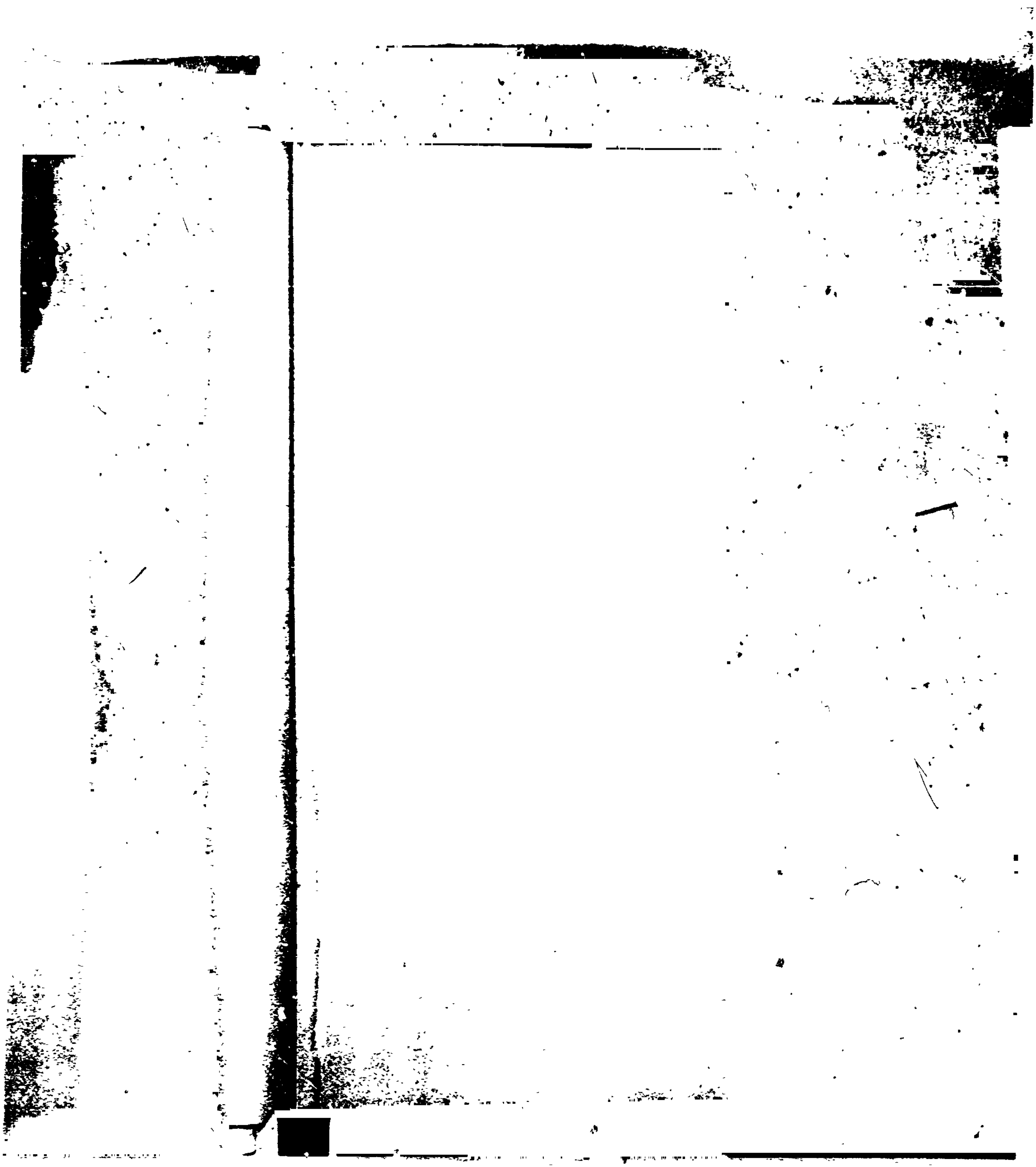
Titania base powder, supplied by the Plasmadyne Corporation and plasma flame-sprayed in a Plasmadyne at Pratt & Whitney Aircraft Division was the coating used for the second test section. Since it had previously been decided that the coating used on the Sunflower section should be the same as the one tested on a SNAP-8 test section, this coating was also used on the third (Sunflower I) test section. The material is essentially titanium dioxide with small amounts of other oxides present.

After 2180 hours of endurance testing, the SNAP-8 test section showed faint cracks in the coating at the top of the tube portion of the test section (Figure 1). After 3490 hours these cracks were plainly visible (Figure 2). A small flake was missing from the tube, at the mid-section, after 6170 hours (Figure 3). After 6840 hours, (Figure 4) the size of this flake had not changed but a second, larger, flake was missing further down the tube and a crack in the coating at the top of the tube was now approximately 1.5 inches long. At the end of the quarter the test section had accumulated 7810 hours of endurance testing.

The Sunflower test section has accumulated 7760 hours of testing with no evidence of cracking or spalling visible.

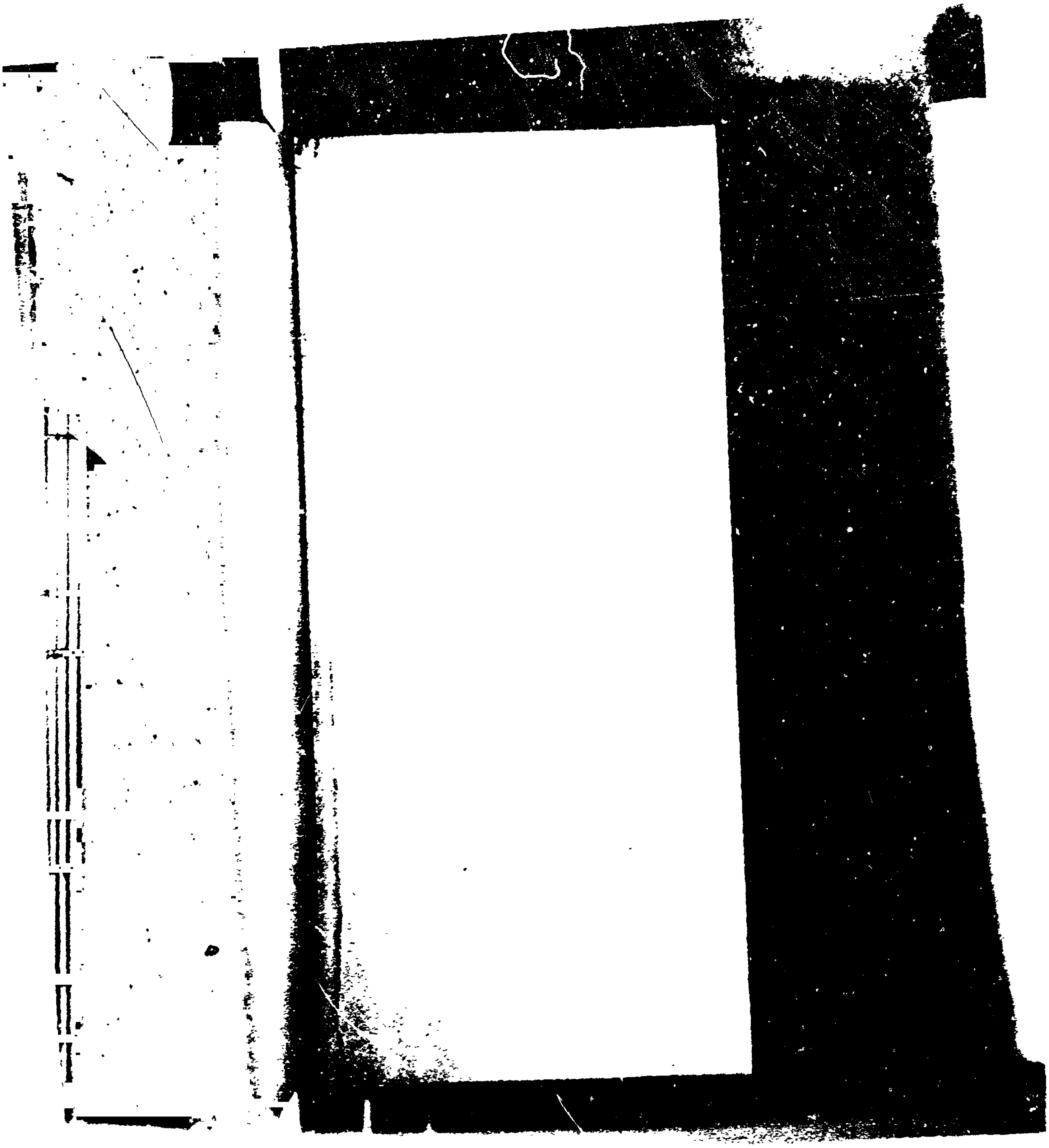
C. Rig No. 4, SNAP-8 Test Section

A mixture of silicon carbide and silicon dioxide is aluminum phosphate-bonded onto this test section. Reports PWA-2012 and 2043 describe the various emittance tests this material has been subjected to. Test time for the specimen totaled 6530 hours at the end of September.



TITANIA BASE COATED SNAP-8 FIN SEGMENT AT 2180 HOURS IN RIG 14134-A. NOTE FAINT CRACKS IN THE SURFACE ON THE UPPER PORTION OF THE TUBE

Figure 1

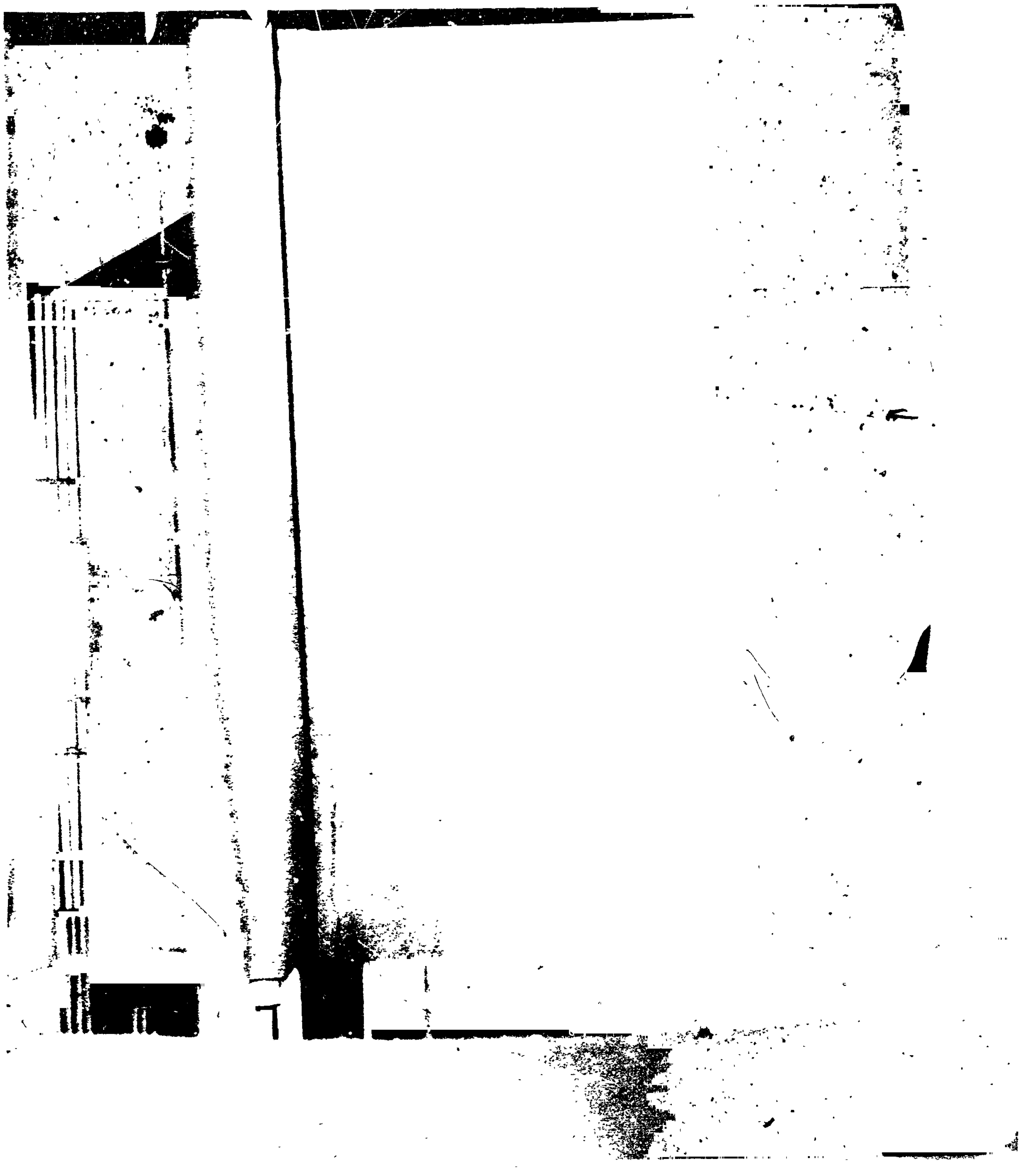


TITANIA BASE COATED SNAP-8 FIN SEGMENT AT 3490 HOURS IN RIG 14134-A. NOTE CRACKS IN THE COATING ON TUBE

Figure 2

TITANIA BASE COATED SNAP-8 FIN SEGMENT AT 6170 HOURS IN RIG 14134-L. NOTE CRACKS IN THE COATING ON TUBE

Figure 3



TITANIA BASE COATED SNAP-8 FIN SEGMENT AT 6840 HOURS IN RIG 14134-L. NOTE LOSS IN COATING ON THE TUBE PORTION OF SPECIMEN

Figure 4

II. EMITTANCE MEASUREMENTS

A. Columbium - 1 Per Cent Zirconium Alloy

Most of the coatings tested during this reporting period were on columbium - 1 per cent zirconium tubes. Two uncoated specimens of columbium - 1 per cent zirconium were tested to establish the emittance characteristics of the substrate material. The first was an "as received" specimen and was tested in the total hemisherical emittance rig only. The second, a polished specimen, was tested in both the spectral and total hemispherical emittance rigs. The total emittance data for the "as received" specimen and for the polished specimen are shown in Figures 5 and 6 respectively.

As may be seen in Figure 6, the total emittance of the polished specimen increased with each successive test run. During this test a leak existed in the rig which resulted in the test being performed in a vacuum of only 2 - 3 x 10^{-6} mm Hg. Although the leak was not serious enough to preclude testing, most of the other tests were conducted with vacuum levels in the 10^{-7} mm Hg range. The fact that a good vacuum could not be maintained even though a large pump was used (400 l/sec) shows that a significant amount of air was being pumped through the chamber during testing. It is suspected that during spectral emittance testing in a vacuum of about 10^{-8} mm Hg the specimen was cleaned up and that subsequently during total emittance testing oxide layers formed on the surface and caused the emittance increases.

Spectral normal emittances of the specimen are shown in Figure 7 for test temperatures of 900°, 1450°, and 2000°F.

TABLE I

Uncoated, As Received Columbium - 1% Zirconium

Run No.	Elapsed Time (Hrs.)	Pressure (mm Hg)	T.C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	0.9	2.4×10^{-6}	310	.114		
	1.7	2.4×10^{-6}	521	.145		
	2.2	2.4×10^{-6}	703	.166		
	2.5	2.3×10^{-6}	903	.187		
	2.8	2.3×10^{-6}	999	.196		
	3.1	2.5×10^{-6}	1100	.219		
	4.3	2.4×10^{-6}	1199	.236		
	4.5	2.4×10^{-6}	1301	.242		
	4.7	2.3×10^{-6}	1399	.255		
	5.0	2.3×10^{-6}	1500	.264	1476	.277
	5.2	2.3×10^{-6}	1602	.274	1565	.295
	5.4	2.2×10^{-6}	1703	.283	1717	.275
	5.7	2.1×10^{-6}	1801	.286	1817	.278
	5.9	2.2×10^{-6}	1904	.291	1917	.285
	6.1	2.2×10^{-6}	2005	.298	2026	.288
	6.4	2.1×10^{-6}	2105	.298	2122	.290
	6.6	2.1×10^{-6}	2203	.305	2221	.297
	6.8	2.1×10^{-6}	2150	.300	2189	.282
	7.0	2.1×10^{-6}	1851	.275	1889	.258
	7.2	2.2×10^{-6}	1549	.247	1569	.238
	7.5	2.2×10^{-6}	1249	.232		
	7.9	2.2×10^{-6}	949	.195		
	8.1	2.4×10^{-6}	653	.161		

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

UNCOATED, AS RECEIVED COLUMBIUM—1% ZIRCONIUM

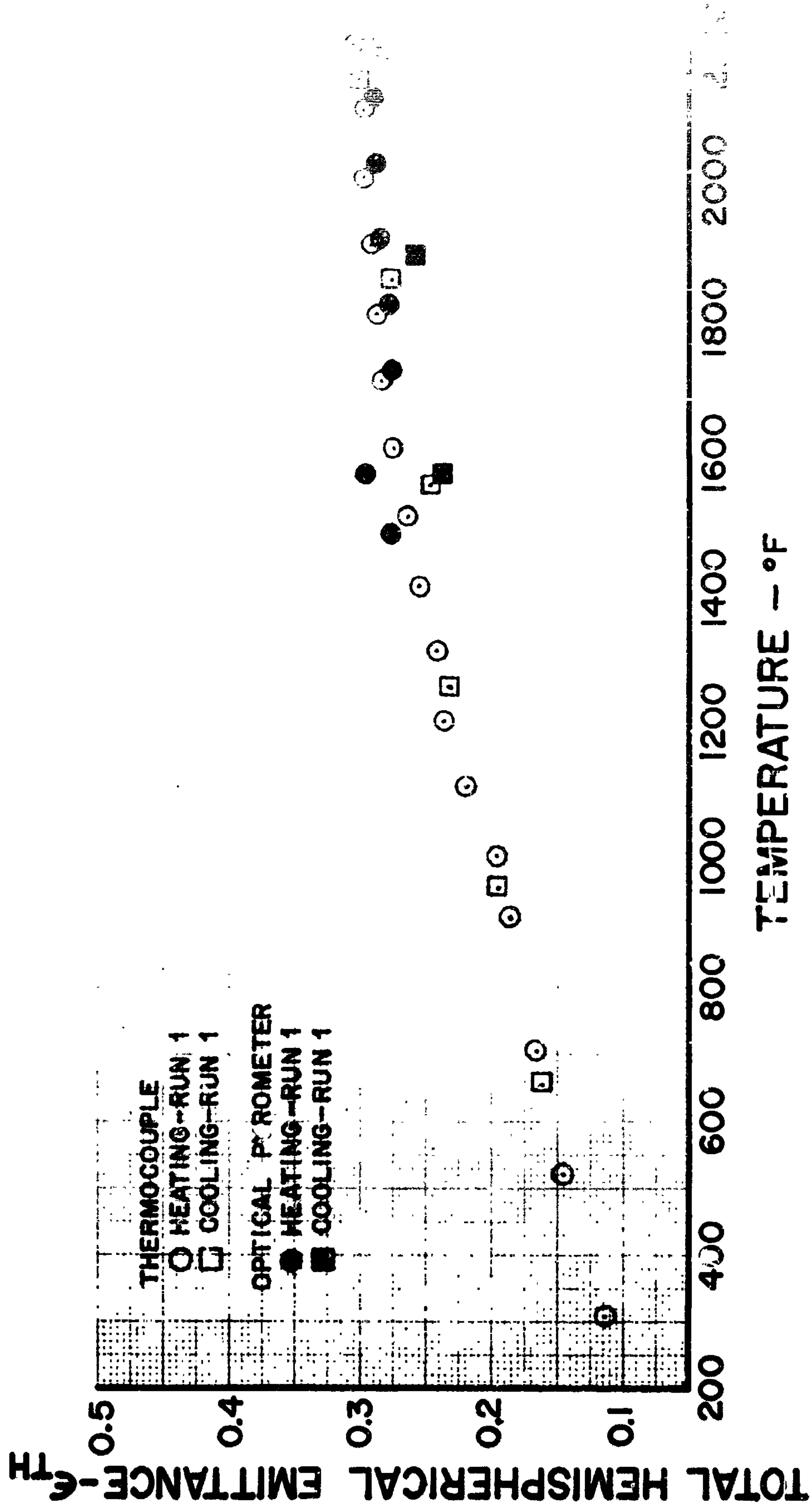


Figure 5

TABLE II

Uncoated, Polished Columbium - 1% Zirconium

Run No.	Elapsed Time (Hrs.)	Pressure (mm Hg)	T.C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	0.2	2.8×10^{-6}	900	.144		
	0.4	2.6×10^{-6}	1176	.147		
	0.6	2.8×10^{-6}	1448	.171		
	0.7	2.7×10^{-6}	1697	.197	1704	.194
	1.0	2.5×10^{-6}	2050	.208	2081	.198
2	1.3	2.5×10^{-6}	902	.154		
	1.5	2.3×10^{-6}	1173	.162		
	1.7	2.4×10^{-6}	1448	.188		
	1.9	2.4×10^{-6}	1687	.212	1683	.213
	2.1	2.2×10^{-6}	2047	.219	2065	.213
3	2.5	2.2×10^{-6}	899	.160		
	2.7	2.1×10^{-6}	1175	.175		
	2.8	2.1×10^{-6}	1454	.192		
	2.9	2.0×10^{-6}	1703	.205		
	3.1	2.0×10^{-6}	2048	.224		
	3.2	2.3×10^{-6}	999	.170		
4	3.7	2.4×10^{-6}	903	.171		
	3.8	2.3×10^{-6}	1176	.177		
	3.9	2.4×10^{-6}	1448	.194		
	4.1	2.4×10^{-6}	1695	.212		
	4.2	2.3×10^{-6}	2048	.195		
	4.4	2.3×10^{-6}	1017	.180		
5	4.6	2.4×10^{-6}	902	.179		
	4.7	2.3×10^{-6}	1176	.184		
	4.8	2.5×10^{-6}	1449	.202		
	5.0	2.5×10^{-6}	1694	.220		
	5.1	3.1×10^{-6}	2044	.241		

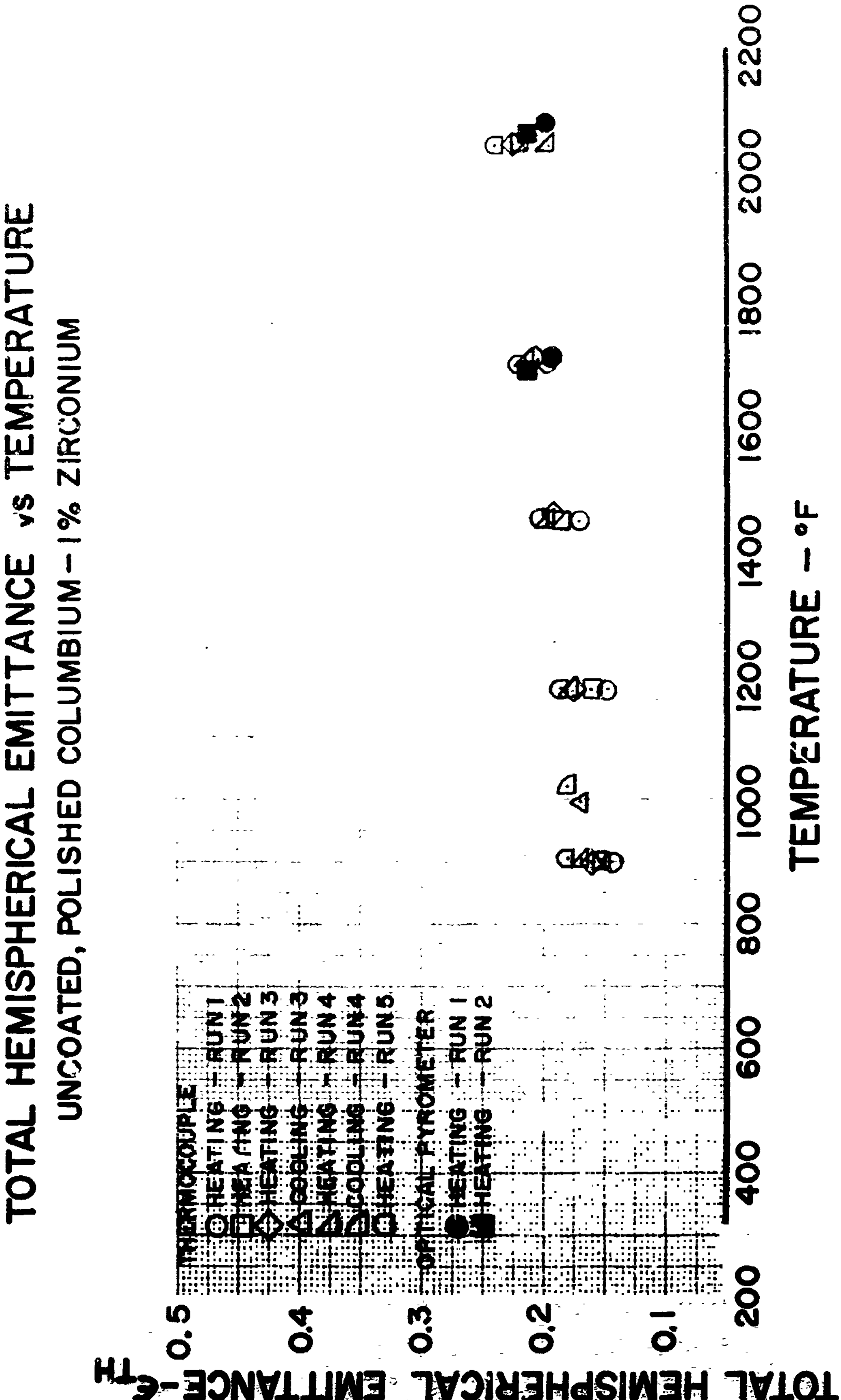

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
UNCOATED, POLISHED COLUMBIUM – 1% ZIRCONIUM
THERMOCOUPLE
HEATING – RUN 1
HEATING – RUN 2
HEATING – RUN 3
COOLING – RUN 3
HEATING – RUN 4
COOLING – RUN 4
HEATING – RUN 5
OPTICAL PYROMETER
HEATING – RUN 1
HEATING – RUN 2
TOTAL HEMISPHERICAL EMITTANCE – ε TH
0.5
0.4
0.3
0.2
0.1
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE – °F

Figure 6

SPECTRAL NORMAL EMITTANCE vs. WAVE LENGTH

UNCOATED, POLISHED COLUMBIUM — 1% ZIRCONIUM

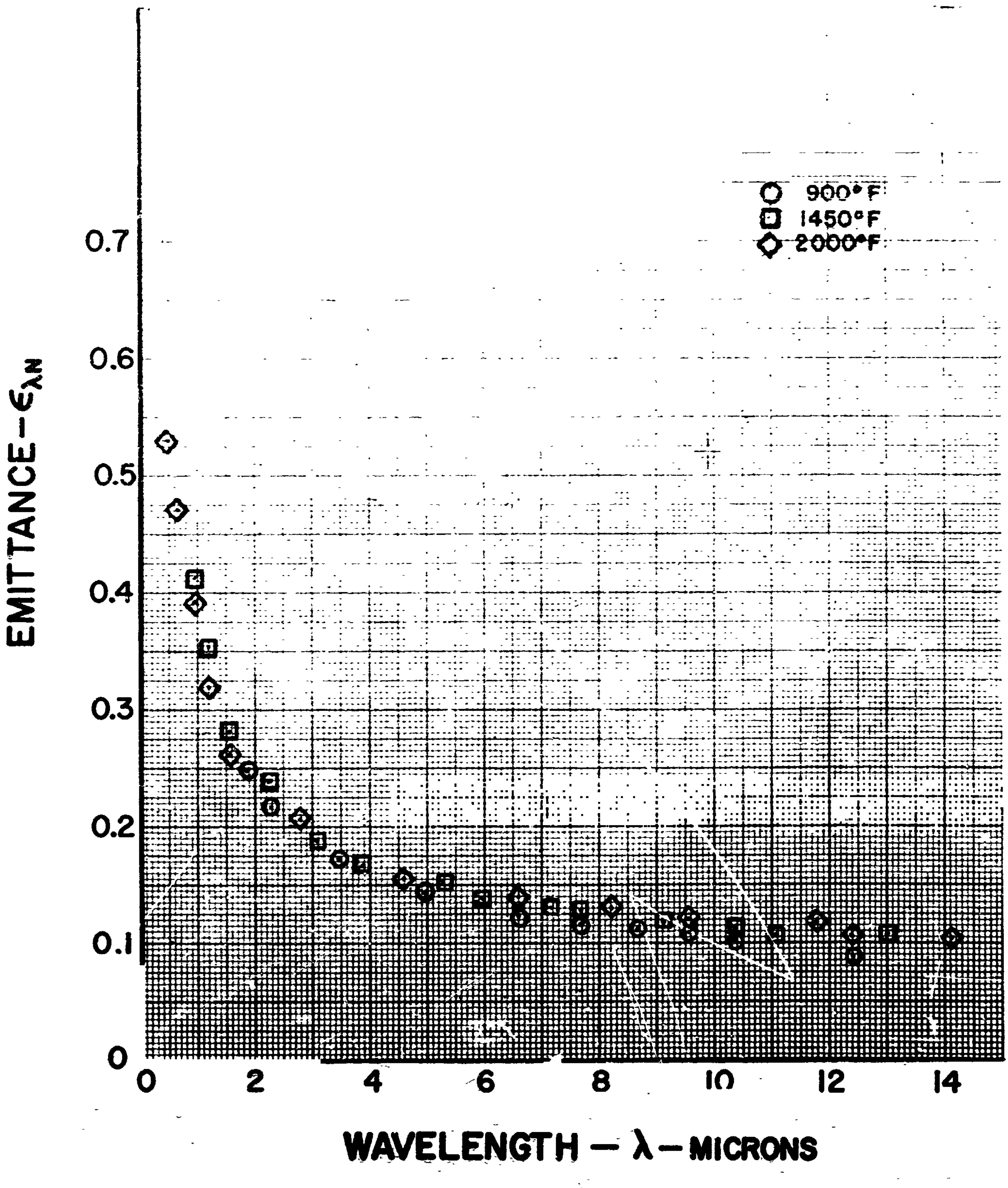


Figure 7

B. Zirconium Silicate

Zirconium silicate coatings ($ZrO_2 \cdot SiO_2$) were applied by plasma-arc spraying and by aluminum-phosphate bonding on a substrate of columbium -1per cent zirconium. The zirconium-silicate powder used all came from a single container and was supplied by the Continental Coatings Corporation (FCZ-11). Three additional specimens were prepared by the Rokide process utilizing Rokide ZS rods which are primarily zirconium silicate. Two of these used a substrate material of columbium - 1 per cent zirconium and the third used a substrate of 310 stainless steel.

1. Plasma-Arc Sprayed Coating on Columbium -1 Per Cent Zirconium

The coatings were white, four mils thick, moderately hard, rough in texture, and were well bonded to the substrate.

a. Total Hemispherical Emittance Test - Total hemispherical emittance was measured from 300°F to 2200°F. As shown in Figure 8, the total emittance decreased from 0.83 at 300°F to 0.51 at 2200°F. The data obtained during the final cooling cycle indicated that a change in the coating had taken place. When the specimen was removed from the rig, small dark spots were noticed which further indicated a change in the coating.

It may be noted that the emittance data calculated using temperatures obtained with an optical pyrometer are somewhat lower than those obtained with the use of a platinum-platinum 10 per cent rhodium thermocouple. It is believed that the optical pyrometer temperatures (obtained by sighting into the black body hole) are the more reliable. The data based on thermocouples during the final portion of the cooling cycle is presented to give some indication of the shape of the emittance curve after the change, although the emittances reported are higher than the true values.

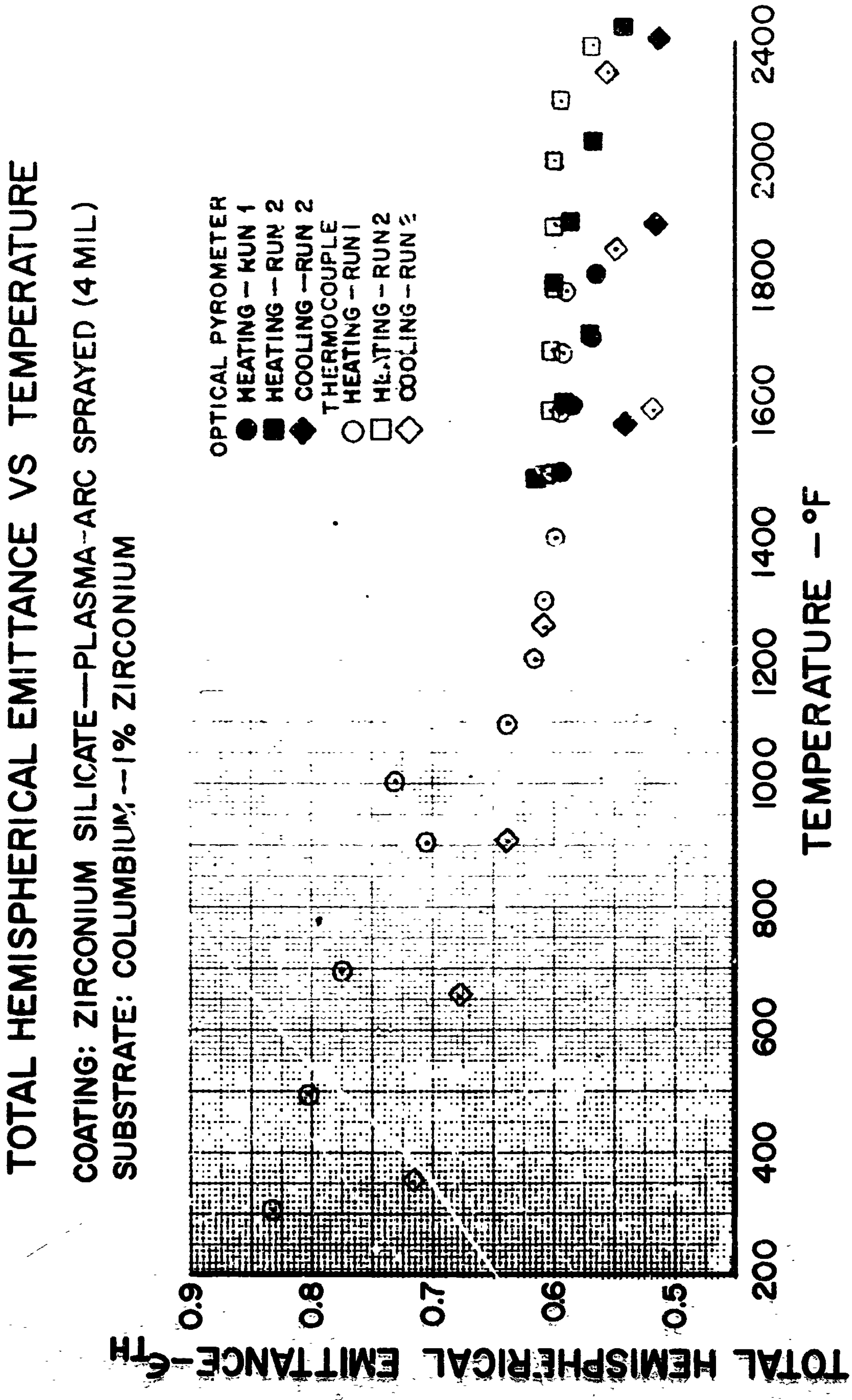
b. Spectral Normal Emissivity Test - Spectral emittance was measured from 1.5 microns to 13.5 microns at 900°F, from 1 micron to 13.5 microns at 1450°F, and from 1 micron to 14 microns at 2000°F. The data are shown in Figure 9 and are typical of that for a white coating, i.e., the spectral emittance is low at short wavelengths, rapidly increases with increasing wavelengths in the 2 to 6 micron range, and then remains high. The dip at 9.5 microns is in the wavelength range where silica emissivity typically shows a minimum.

TABLE III
Coating: Zirconium Silicate - Plasma
Arc Sprayed
Substrate: Columbium - 1% Zirconium
4.0 Mil Coating

Run No.	Elapsed Time (Hrs)	Pressure (mm Hg)	T.C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	0.5	2.2×10^{-6}	307	.832		
	1.1	2.4×10^{-6}	497	.801		
	1.6	2.1×10^{-6}	698	.775		
	2.0	2.2×10^{-6}	902	.705		
	3.8	2.1×10^{-6}	1001	.731		
	4.2	2.1×10^{-6}	1099	.639		
	4.4	2.1×10^{-6}	1200	.615		
	4.7	2.2×10^{-6}	1299	.608		
	5.3	2.1×10^{-6}	1397	.599		
	6.7	2.1×10^{-6}	1500	.602	1507	.593
	6.9	2.0×10^{-6}	1600	.594	1610	.583
	7.1	2.1×10^{-6}	1699	.591	1720	.569
	7.2	2.2×10^{-6}	1799	.589	1824	.564

Heating Current Off; Vacuum Maintained

Run No.	Elapsed Time (Hrs)	Pressure (mm Hg)	T.C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
2	7.6	2.3×10^{-6}	1500	.607	1495	.614
	7.9	2.2×10^{-6}	1601	.601	1610	.591
	8.1	2.2×10^{-6}	1700	.600	1728	.569
	8.4	2.3×10^{-6}	1800	.600	1802	.598
	8.6	2.3×10^{-6}	1901	.597	1910	.585
	8.9	2.4×10^{-6}	2008	.597	2040	.567
	9.2	2.2×10^{-6}	2104	.592	2152	.549
	9.5	2.0×10^{-6}	2195	.566	2225	.541
	9.8	1.9×10^{-6}	2150	.554	2202	.512
	11.2	1.9×10^{-6}	1866	.548	1903	.514
	11.5	2.0×10^{-6}	1601	.518	1580	.539
	11.7	2.2×10^{-6}	1252	.607		
	12.3	2.1×10^{-6}	906	.639		
	12.6	2.6×10^{-6}	653	.677		
	13.0	2.0×10^{-6}	352	.717		



TOTAL HEMISPHERICAL EMITTANCE VS TEMPERATURE
COATING: ZIRCONIUM SILICATE—PLASMA-ARC SPRAYED (4 MIL)
SUBSTRATE: COLUMBIUM—1% ZIRCONIUM
OPTICAL PYROMETER
HEATING — RUN 1
HEATING — RUN 2
COOLING — RUN 2
THERMOCOUPLE
HEATING — RUN I
HEATING — RUN 2
COOLING — RUN 2
0.9
0.8
0.7
0.6
0.5
TOTAL HEMISPHERICAL EMITTANCE — ϵ_{TH}
200
400
600
800
1000
1200
1400
1600
1800
2000
2400
TEMPERATURE — °F

Figure 8

SPECTRAL NORMAL EMITTANCE VS. WAVELENGTH

COATING: ZIRCONIUM SILICATE—PLASMA–ARC SPRAYED (4 MIL)
SUBSTRATE: COLUMBIUM—1% ZIRCONIUM

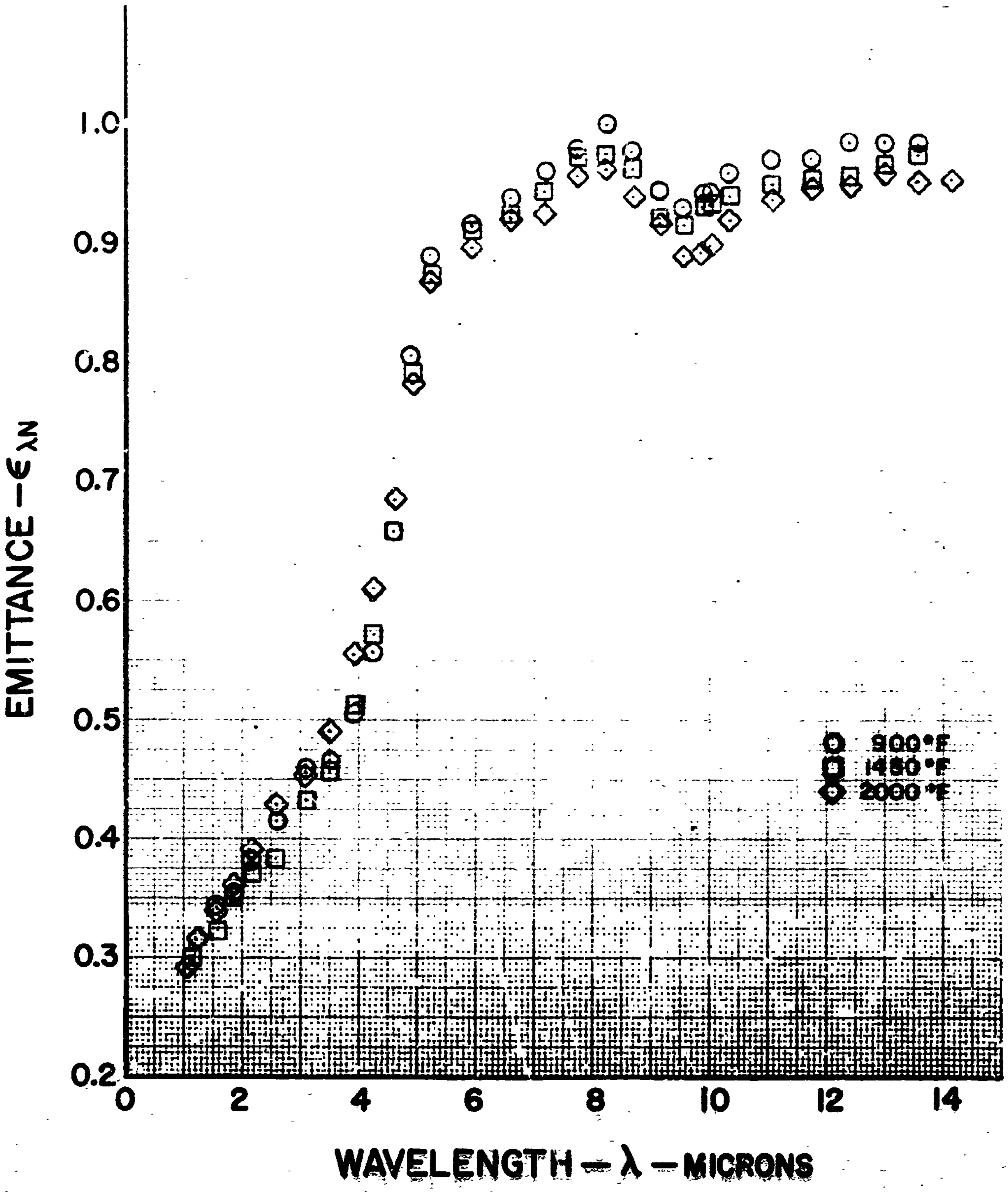


Figure 9

2. Aluminum-Phosphate-Bonded Coating on Columbium-1 Per Cent Zirconium

a. Total Hemispherical Emittance Test - The same zirconium silicate powder was used for this specimen as for the plasma arc-sprayed coatings discussed above. The specimen tested in the total emittance rig had a 4-mil thick coating which was white, very soft, and poorly bonded to the substrate.

Total emittance was measured from 300°F to 1850°F. As may be seen in Figure 10 , the emittance decreased from 0.75 at 300°F to about 0.39 at 1850°F. It was noticed that the specimen was whiter after the test than before.

Comparison of Figures 8 and 10 shows that for temperatures ranging up to approximately 1400°F the emittance values of the phosphate-bonded coating are about 10 per cent lower than those of the plasma arc-sprayed coating. Above 1400°F the differences increase.

As in the tests discussed previously, at elevated temperatures there existed considerable discrepancy between temperature measurements obtained with an optical pyrometer and those obtained with a platinum-platinum 10 per cent rhodium thermocouple. The size of this discrepancy indicated that the platinum-platinum 10 per cent rhodium thermocouple was completely unreliable for the high-temperature portion of this test. A chromel-alumel thermocouple, used as a reliability check, provided temperature data in reasonably good agreement with the optical pyrometer and therefore data based on the chromel-alumel thermocouple is reported rather than that based on the platinum-platinum 10 per cent rhodium thermocouple.

b. Spectral Normal Emittance Test - The specimen for spectral normal emittance testing was prepared at the same time and had the same external characteristics as the specimen for total hemispherical emittance testing. The 4-mil thick coating became whiter during testing.

TABLE IV

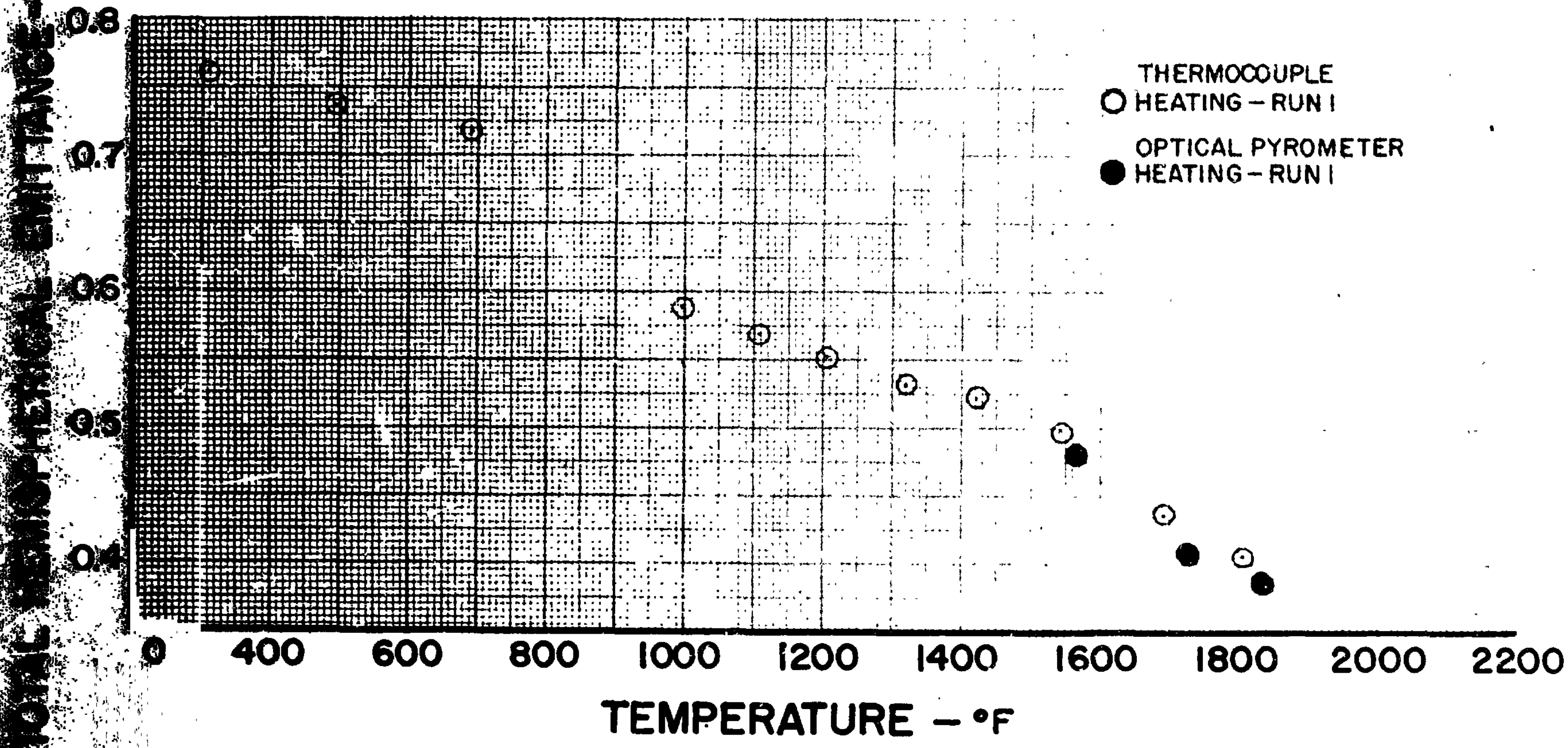
Coating: Zirconium Silicate - Aluminum Phosphate Bonded
Substrate: Columbium - 1% Zirconium

4 Mil Coating

Run No.	Elapsed Time (Hrs.)	Pressure (mm Hg)	T. C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	0.6	3.1×10^{-7}	307	.761		
	0.9	2.7×10^{-7}	497	.737		
	1.3	2.9×10^{-7}	690	.716		
	4.2	2.7×10^{-7}	999	.589		
	4.5	2.9×10^{-7}	1105	.568		
	4.7	5.7×10^{-7}	1206	.552		
	4.9	1.2×10^{-6}	1321	.531		
	5.1	2.7×10^{-6}	1420	.523		
	5.2	1.1×10^{-5}	1546	.498	1563	.482
	5.5	1.1×10^{-6}	1694	.436	1728	.409
	5.9	3.7×10^{-7}	1808	.407	1837	.387

TOTAL HEMISPHERICAL EMITTANCE VS. TEMPERATURE

COATING: ZIRCONIUM SILICATE — ALUMINUM PHOSPHATE BONDED (4 MIL)
SUBSTRATE: COLUMBIUM — 1% ZIRCONIUM



Spectral emittance was measured from 1.5 to 12.5 microns at 900°F from 1 to 13.5 microns at 1400°F, and from 0.5 to 13.5 microns at 2000°F. The spectral emittance data are shown in Figure 11.

It is to be noted that at short wave lengths the spectral emittance was higher at 900°F and 1450°F than would have been expected for a white coating, but that when the test temperature was raised to 2000°F the emittance dropped to a more reasonable level. The emittance drop is consistent with the increased whiteness of the specimen after testing.

Comparison of Figures 9 and 11 reveals several differences between the curve shapes for the two specimens. The curve for the aluminum phosphate-bonded specimen appears similar to that of the plasma arc-sprayed specimen but is shifted 1 to 2 microns to the right. The shift appears most noticeably at the dip which has a minimum at 9.5 microns for the plasma arc-sprayed specimen and at about 11 microns for the aluminum phosphate-bonded specimen. It is further noticed that the curve for the aluminum phosphate-bonded specimen shows a dip at about 6 microns which does not appear on the curve for the plasma arc-sprayed specimen.

SPECTRAL NORMAL EMITTANCE VS. WAVELENGTH

COATING: ZIRCONIUM SILICATE--ALUMINUM-PHOSPHATE BONDED (4 MIL)

SUBSTRATE: COLUMBIUM—1% ZIRCONIUM

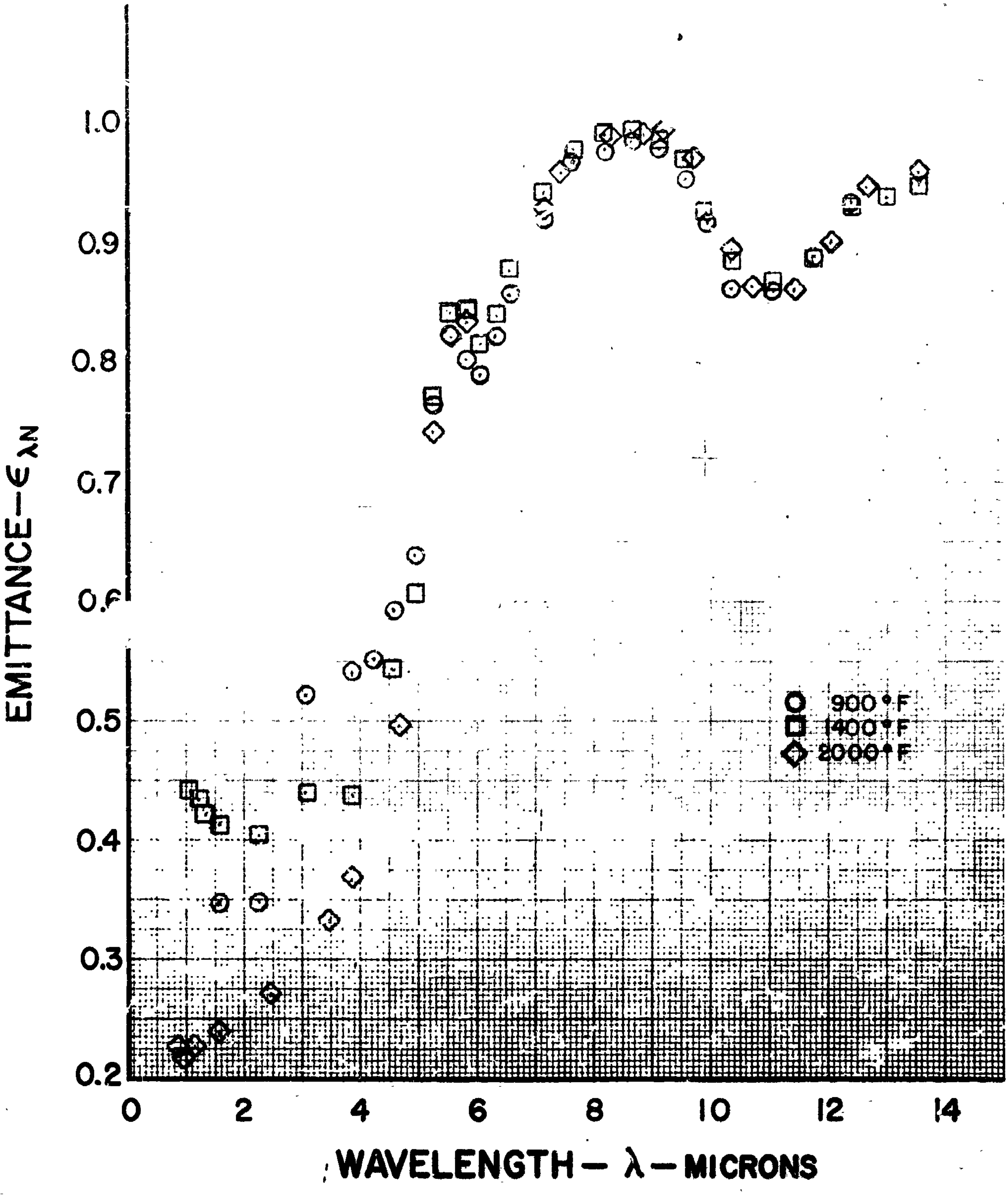


Figure 11

3. Rokide ZS Coatings

Rokide rods, obtained from the Norton Company, were used to apply coatings to both type 310 stainless steel and to columbium - 1 per cent zirconium substrates. A typical analysis of these rods as determined by the Norton Company is as follows: 64.12 per cent ZrO_2, 33.22 per cent SiO_2, 1.42 per cent Al_2O_3, and 0.97 per cent ($CaO + TiO_2 + Fe_2O_3 + NaO_2$). Both types of specimens were tested to determine possible effects of the two different substrates. The coatings were white in color and had a rough but glassy appearance.

a. Rokide ZS Coating on 310 Stainless Steel - Total Hemispherical Emittance Test - The 4-mil thick coating was very hard with a fair coating-substrate bond. As shown in Figure 12, the total hemispherical emittance decreased from 0.65 at 1000°F to approximately 0.53 at 2150°F, then increased to approximately 0.56 at 2200°F. When the specimen was removed from the rig it was noted that the color had changed from white to grey, indicating that the emittance increase at 2200°F probably resulted from a change in the coating. It was also observed that a considerable amount of the coating had spalled off the tube which probably resulted from the large difference in coefficients of thermal expansion between the coating and the substrate.

b. Rokide ZS Coating on Columbium - 1 Per Cent Zirconium - Total Hemispherical Emittance - The 5-mil thick coating was hard and wellbonded to the substrate. As shown in Figure 13, the total hemispherical emittance decreased from 0.75 at 500°F to 0.6 at 1500°F and then increased to approximately 0.68 at 2200°F. The data agreed fairly well with those for Rokide ZS on 310 stainless steel between 1000°F and 1500°F. At this temperature the emittance started to increase, apparently the result of a change in the coating. The color of this specimen also changed to grey. This is consistent with the similar changes in emittance of the two specimens. No spalling of the coating occurred. None was expected since the difference in coefficients of thermal expansion between the coating and this substrate is small compared to that of the previous specimen.

TABLE V

Coating: Rokide ZS
Substrate: Stainless Steel 310

4.0 Mil Coating

Run No.	Elapsed Time (Hrs.)	Pressure (mm Hg)	T. C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	4.2	3.2×10^{-7}	1001	.641		
	4.3	3.3×10^{-7}	1102	.630		
	4.5	3.4×10^{-7}	1201	.623		
	4.7	3.8×10^{-7}	1302	.611		
	4.8	3.2×10^{-7}	1401	.586		
	5.1	2.9×10^{-7}	1502	.577	1515	.562
	5.2	5.2×10^{-7}	1601	.568	1628	.539
	5.4	1.2×10^{-6}	1700	.556		
	5.6	2.8×10^{-6}	1796	.552	1808	.541
	5.8	6.2×10^{-6}			1910	.537
	5.9	1.1×10^{-5}			2033	.528
	6.1	1.1×10^{-5}			2162	.527
	6.2	1.0×10^{-5}			2204	.561

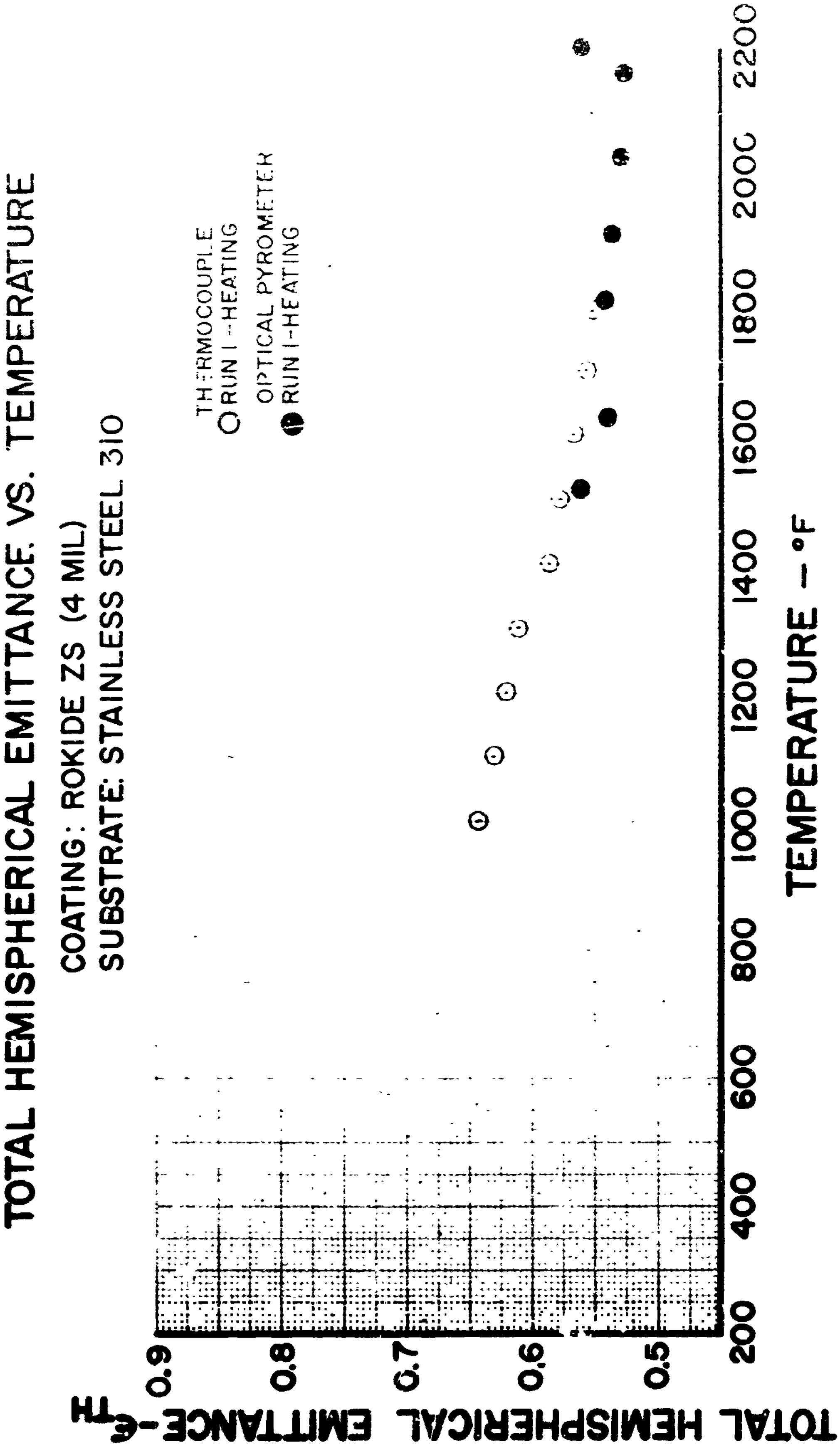

TOTAL HEMISPHERICAL EMITTANCE VS. TEMPERATURE
COATING: ROKIDE ZS (4 MIL)
SUBSTRATE: STAINLESS STEEL 310
THERMOCOUPLE
RUN 1--HEATING
OPTICAL PYROMETER
RUN 1-HEATING
TOTAL HEMISPHERICAL EMITTANCE-ϵ_{TH}
0.9
0.8
0.7
0.6
0.5
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE — °F

Figure 12

[illegible]

[illegible] Zr
Substrate: Columbium - 1% Zirconium

[illegible]

Run No.	Elapsed Time (Hrs)	Pressure (mm Hg)	T. C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	1.2	[illegible]	497	.752		
	1.4	[illegible]	[illegible]	.731		
	1.8	[illegible]$\times10^{-7}$	694	.689		
	2.0	5.3x10^{-7}	[illegible]	.672		
	2.2	7.[illegible]x10^{-7}	1101	.655		
	2.3	8.1x10^{-7}	1199	.640		
	2.5	9.4x10^{-7}	1300	.630		
	2.7	7.9x10^{-7}	1400	.613		
	2.9	8.8x10^{-7}	1496	.599		
	3.1	5.8x10^{-7}	1602	.603	1606	.598
	3.7	3.6x10^{-7}	1793	.602	1796	.599
	3.9	5.0x10^{-7}			1915	.604
	4.1	5.3x10^{-7}			2045	.638
	4.2	5.1x10^{-7}			2160	.681
	4.4	4.8x10^{-7}			2232	.679
	4.6	3.9x10^{-7}			2195	.672
	4.8	3.1x10^{-7}			1867	.610
	4.9	3.2x10^{-7}			1578	.551

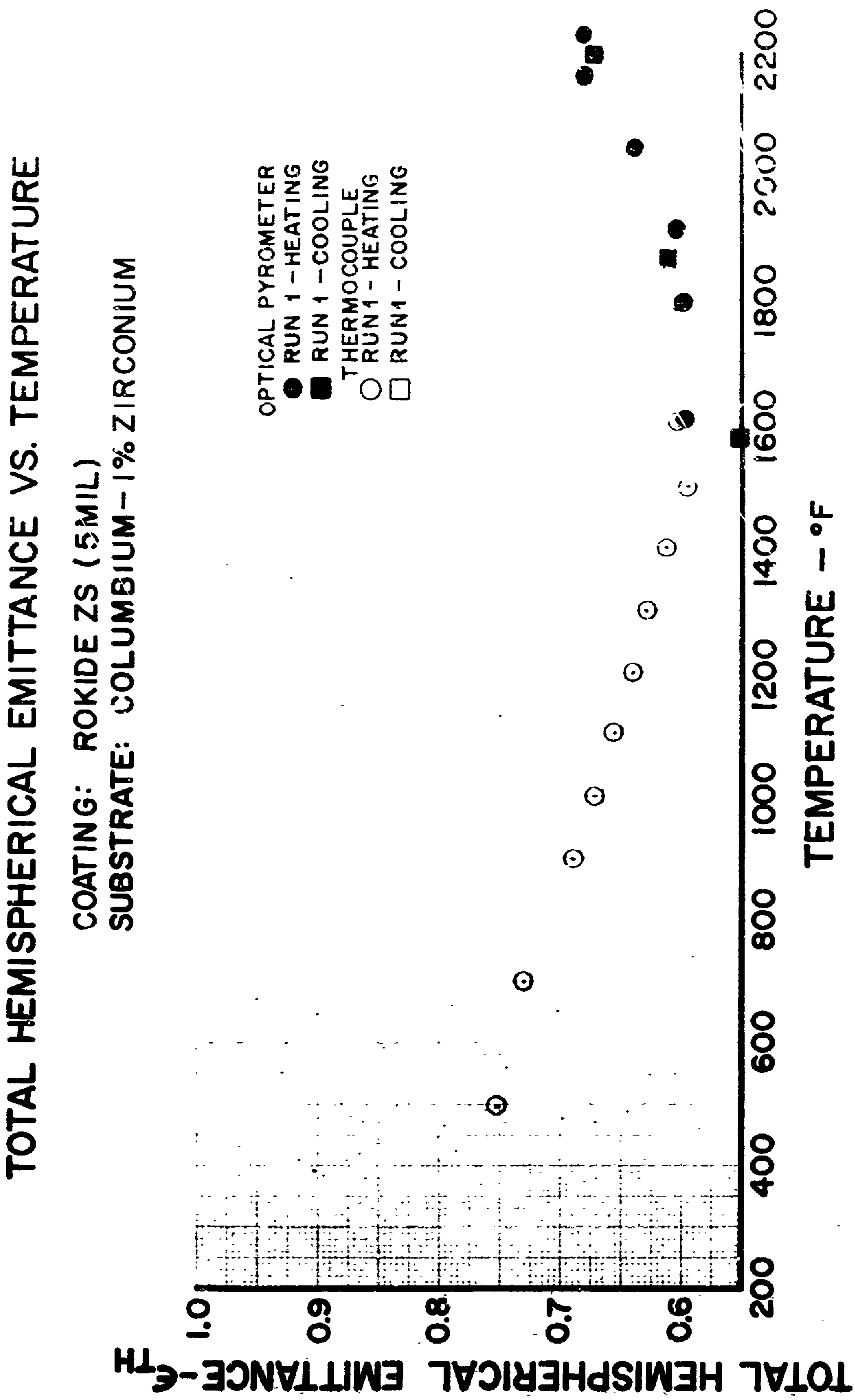

TOTAL HEMISPHERICAL EMITTANCE VS. TEMPERATURE
COATING: ROKIDE ZS (5MIL)
SUBSTRATE: COLUMBIUM–1% ZIRCONIUM
OPTICAL PYROMETER
RUN 1 –HEATING
RUN 1 –COOLING
THERMOCOUPLE
RUN1 - HEATING
RUN1 - COOLING
TOTAL HEMISPHERICAL EMITTANCE-ε TH
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE — °F

Figure 13

c. <u>Rokide ZS Coating on Columbium - 1 Per Cent Zirconium - Endurance Test</u> - The physical characteristics of this specimen were identical to those of the specimen discussed previously. The specimen was maintained at a nominally constant temperature of 1850°F for 280 hours. During the early portion of the test, the optical pyrometer readings obtained were found to be in error and a platinum-platinum 10 per cent rhodium thermocouple was used for temperature measurement until the source of the error was found. The optical pyrometer and the thermocouple were both used for the remainder of the test, but by this time a discrepancy of about 25 to 30°F existed between the two instruments. This results in a corresponding discrepancy in computed emittance of about 5 to 6 per cent. During the portion of the test in which the pyrometer was not used (95 hours) the emittance increased from 0.65 to 0.75, using thermocouple temperature indications and to 0.79, using optical pyrometer temperature indications. A gradual increase to an emittance of 0.83, (based on optical pyrometer temperature measurements) occurred during the next 170 hours. The difference between the emittance values calculated using the thermocouple and the pyrometer remained about 5 to 6 per cent throughout the test. During the remaining 50 hours of testing no further changes took place.

Since an increase in emittance had occurred and since previous tests with Rokide ZS on columbium - 1 per cent zirconium substrates have indicated difficulty in obtaining reliable temperature data with platinum-platinum 10 per cent rhodium thermocouples, it was not possible to determine whether the indicated emittance values at the start of the test were reliable. For this reason, another specimen from the same batch was tested for about 75 hours at the same temperature. The emittance at the start of the second test based on optical pyrometer temperature measurements was 0.65 and increased to about 0.80 during the 75 hours of testing. These results, which were almost identical to those of the first endurance test, confirmed the reliability of the optical pyrometer in the first test, and also indicated that the thermocouple was reliable at the beginning of the endurance test.

The data for these two endurance tests are shown in Figure 14. (During the portion of the first test in which emittance was not changing rapidly, average daily values are shown for days when more than one point was taken.) Emittance data obtained as the specimen was heated to the endurance test temperature are shown in Figure 15.

In each case, when the specimen was removed from the test rig, it was noted that the coating color had changed from white to grey.

4. Zirconium Silicate - General Remarks

X-ray diffraction analyses of the Rokide and plasma arc-sprayed specimens before testing showed cubic zirconia (ZrO_2) to be the principle phase present. Apparently zirconium silicate decomposed at the very high spraying temperatures and the silica thus formed was in a glassy state.[1,2] Spectrographic analysis confirmed the presence of large amounts of silicon in addition to the zirconium. The plasma arc-sprayed specimen used for total hemispherical emittance testing and the Rokide ZS coating on 310 stainless steel were examined after testing and the spectrographic analysis showed no significant change in the elements present. X-ray diffraction showed the presence of both monoclinic and cubic zirconia. The change in crystal structure of the Rokide coatings may be attributed to the presence of magnesium in the coating which, when present as an oxide, can cause zirconia to have either of these structures at room temperature. Spectrographic analysis of the powder used for plasma-arc spraying did not show the presence of magnesium however. Once the powder has been stabilized and converted to the cubic structure, it would not be expected that it would change back to the monoclinic form. No explanation can therefore be given at this time as to why the plasma arc-sprayed powder changed from cubic to monoclinic zirconia.

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1 I. E. Campbell, High Temperature Technology, New York, John Riley & Sons, Incorporated, 1956, P. 82

2 E. Ryshkewitch, Oxide Ceramics, Physical Chemistry and Technology, New York, Academy Press, 1960, P. 399.

TABLE VII

Coating : Rokide ZS
Substrate : Columbium - 1% Zirconium
5 Mil Coating

Run No.	Elapsed Time (Hrs)	Pressure (mm H_g)	Temp (°F)	ϵ_{TH}
1	436.3	1.7×10^{-8}	300	.910
	436.6	8.3×10^{-8}	400	.880
	437.1	2.5×10^{-7}	500	.845
	437.9	4.7×10^{-7}	598	.820
	438.3	1.4×10^{-6}	700	.800
	438.7	2.8×10^{-6}	801	.766
	439.0	2.9×10^{-6}	902	.737
	439.7	7.5×10^{-7}	1002	.715
	440.1	2.5×10^{-6}	1102	.689
	440.4	3.3×10^{-6}	1201	.671
	440.9	2.4×10^{-6}	1298	.649
	441.6	2.3×10^{-6}	1402	.647
	441.9	2.8×10^{-6}	1451	.646

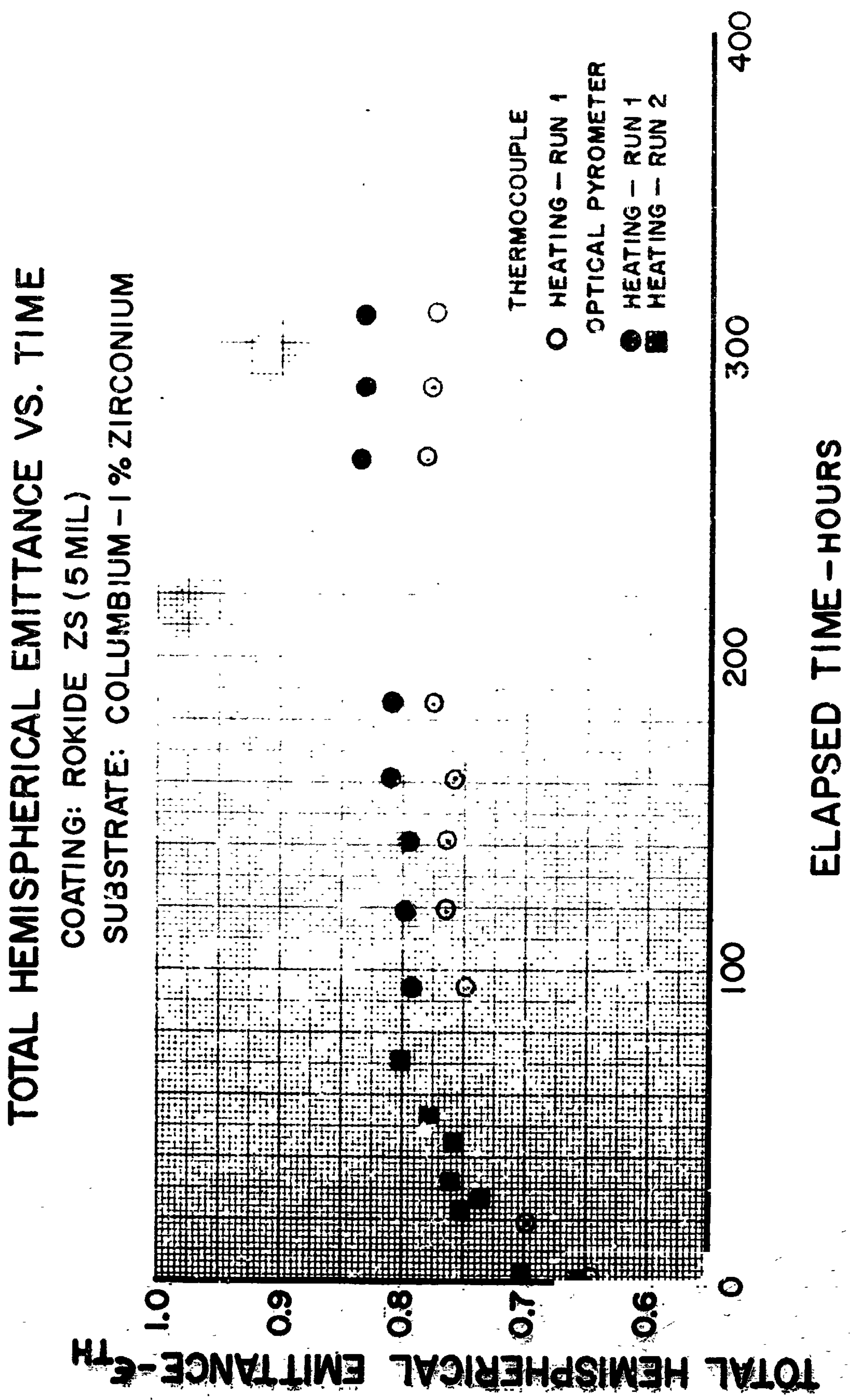
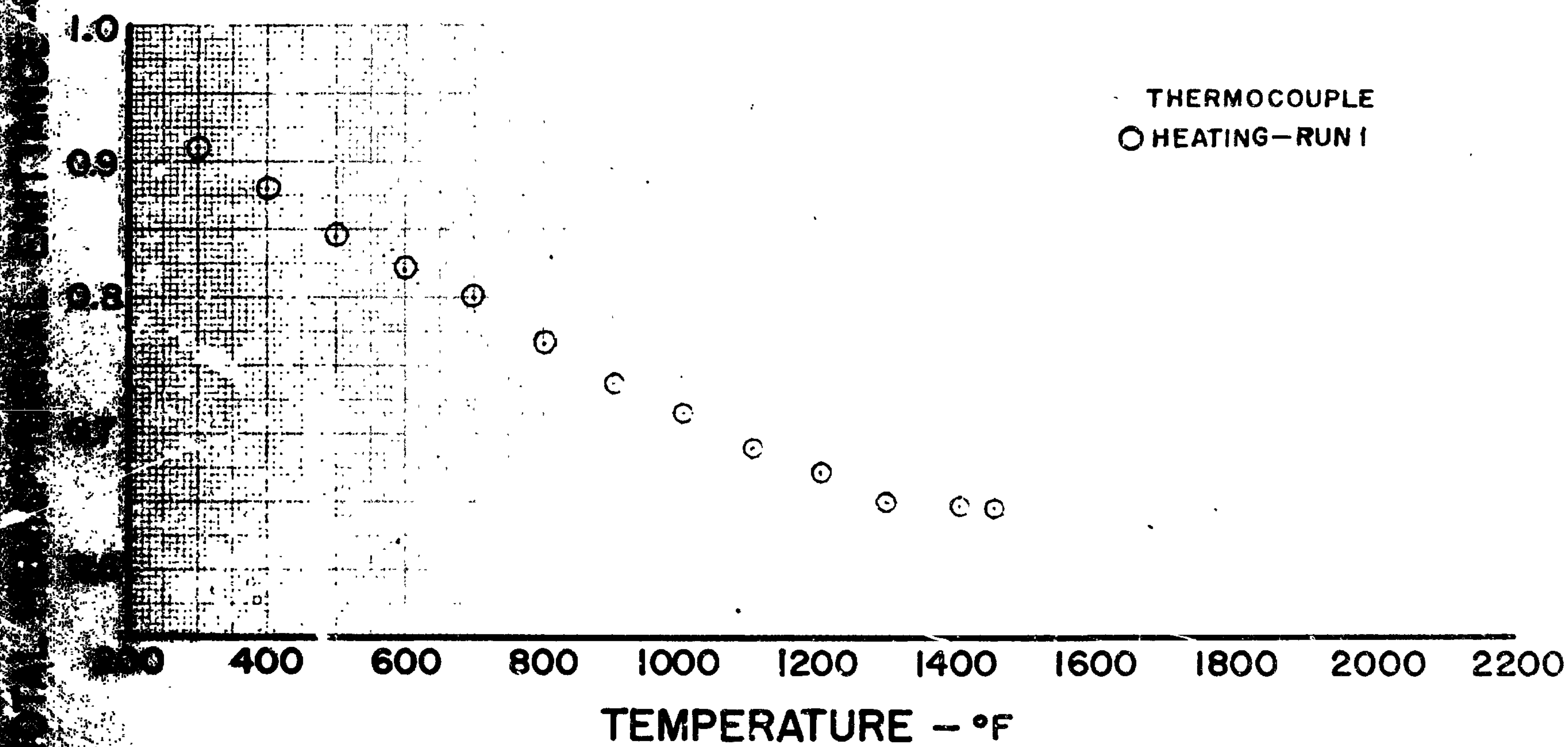

TOTAL HEMISPHERICAL EMITTANCE VS. TIME
COATING: ROKIDE ZS (5 MIL)
SUBSTRATE: COLUMBIUM - 1 % ZIRCONIUM
THERMOCOUPLE
HEATING - RUN 1
OPTICAL PYROMETER
HEATING - RUN 1
HEATING - RUN 2
TOTAL HEMISPHERICAL EMITTANCE - ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
0
100
200
300
400
ELAPSED TIME - HOURS

Figure 14

TOTAL HEMISPHERICAL EMITTANCE VS. TEMPERATURE

COATING: ROKIDE ZS (5 MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM



It has been noted that all of these specimens showed changes in emittance as well as a change in color from white to grey. Whether or not these changes are related to the indicated changes in the structure of zirconia cannot be determined at the present time. If this material is ever considered as a spacecraft radiator coating (which may be justified by the moderately high emittance levels obtained in the latter portion of the endurance testing) additional material analysis techniques will be employed.

An unexplainable factor observed during the test of a plasma sprayed zirconia specimen conducted in 1960 (PWA-1863, P. 149) was what was considered an unusually high and relatively constant emittance level when compared to published values of emittance for zirconia. The specimen tested in 1960, however, was characterized by the same grey color as the recently tested Rokide ZS specimens rather than by the usual color of zirconia. Its emissivity therefore compares with recent test values rather than with values previously published.

X-ray diffraction of the aluminum phosphate-bonded coatings before and after testing showed zirconium silicate ($ZrO_2 \cdot SiO_2$) to be the only crystalline phase present. This is consistent with the lack of a color change during testing of these specimens.

C. Magnesium Aluminate (Spinel)

Coatings of magnesium aluminate ($MgO \cdot Al_2O_3$) were applied to columbium - 1 per cent zirconium tubes by the Rokide process using Rokide MA rods. The composition of typical Rokide MA rods is described by the supplier, The Norton Co. as follows: 66.8 per cent Al_2O_3, 29.5 per cent MgO, 2.[illegible] per cent SiO_2, 0.69 per cent Cr_2O_3 and 0.08 per cent (Fe_2O_3 + TiO_2 + N_aO_2). The well bonded coatings, 5 mils thick, were white, very hard, and had a rough, glossy appearance. Two specimens were tested, one in the total hemispherical emittance rig and the other in the spectral emittance rig. Both were prepared at the same time using rods from the same batch.

1. Total Emittance Test

Total emittance was measured from 300°F to 2200°F and the data are shown in Figure 16 . During the heating cycle, the emittance varied from approximately 0.8 at 300°F to 0.48 at 1800°F and then slowly increased to 0.55 at 2200°F. Maintaining the specimen at this temperature for 30 minutes produced a further increase in emittance to 0.60. Emittances measured as the specimen was cooled indicated that the emittance had increased at all temperatures so it was suspected that the increase resulted from a change in the coating. When the specimen was removed from the rig, it was noted that the color of the specimen had changed from white to grey and that the surface now had a matte appearance as contrasted to its glossy appearance before testing. Although a change in the coating was indicated by the increase in emittance level and further substantiated by the change in the color of the coating, x-ray diffraction analysis showed magnesium-aluminate ($MgO \cdot Al_2O_3$) to be the only crystalline phase present both before and after testing.

2. Spectral Emittance Test

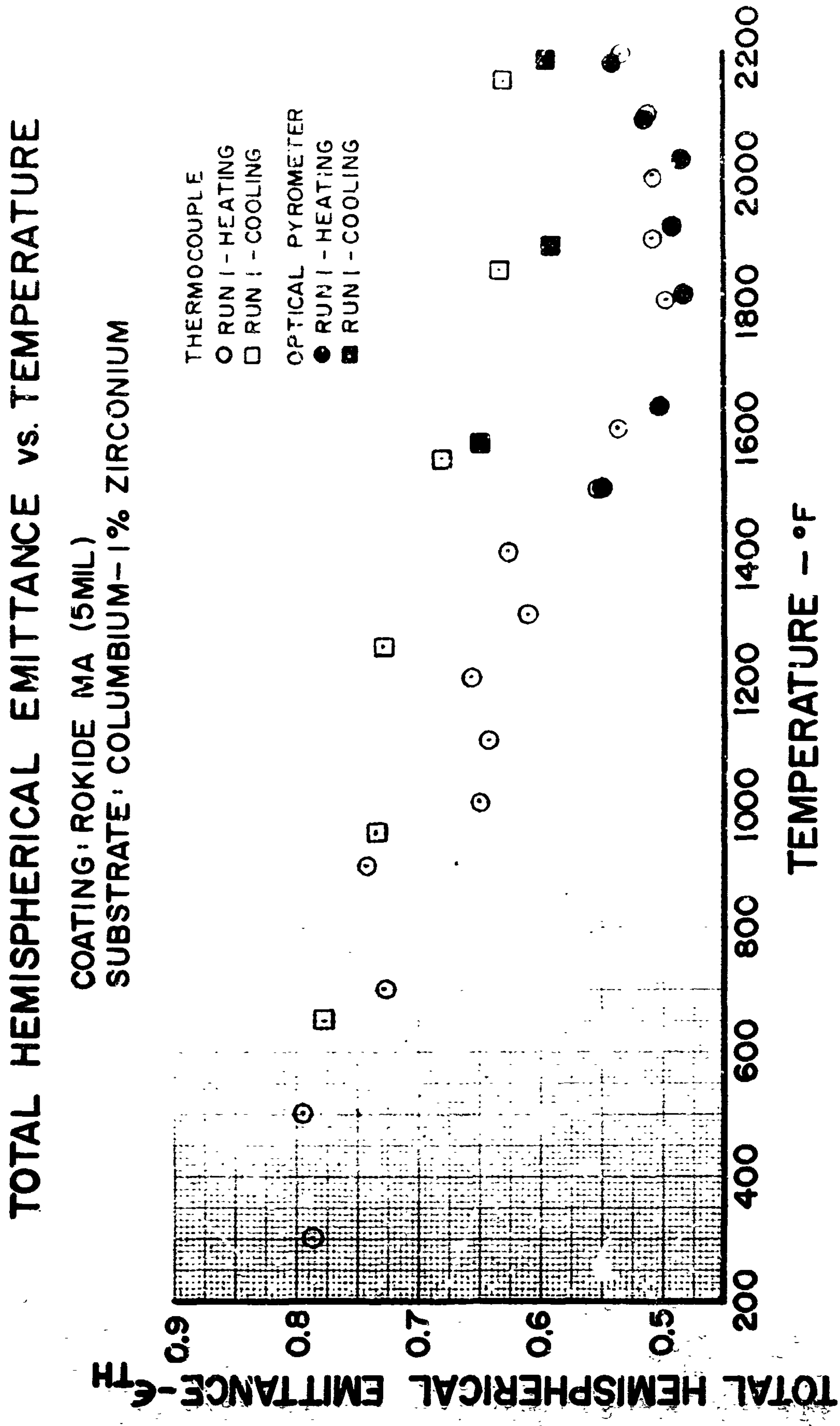
During testing the 5-mil, white coating turned grey-white and the hardness changed from very hard to hard but brittle. The coating remained well bonded to the substrate.

Spectral emittance was measured from 1.5 to 12.4 microns at 900°F, from 1.1 to 13.5 microns at 1450°F, and from 0.45 to 13.5 microns at 2000°F. The spectral emittance data appears in Figure 17 . An interesting phenomenon is the local maximum at approximately 3 microns which is very pronounced at 900°, still exists at 1450°F, and does not appear at all at 2000°F (see inset - Figure 17).

TABLE VIII

Coating: Rokide MA
Substrate: Columbium -1% Zirconium
5.0 Mil Coating

Run No.	Elapsed Time (Hrs.)	Pressure (mm. Hg)	T. C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	2.2	2.0×10^{-7}	303	.787		
	2.6	2.0×10^{-7}	501	.735		
	3.1	2.0×10^{-7}	700	.727		
	3.6	2.0×10^{-7}	901	.742		
	4.6	2.0×10^{-7}	1000	.650		
	4.9	2.4×10^{-7}	1100	.643		
	5.2	2.6×10^{-7}	1201	.656		
	5.5	3.0×10^{-7}	1300	.610		
	5.8	3.3×10^{-7}	1400	.626		
	6.2	2.8×10^{-7}	1500	.552	1502	.550
	6.6	2.4×10^{-7}	1599	.537	1633	.503
	7.3	2.2×10^{-7}	1800	.497	1816	.483
	7.6	1.9×10^{-7}	1899	.509	1920	.491
	7.9	2.2×10^{-7}	1998	.509	2028	.485
	8.1	2.8×10^{-7}	2096	.512	2095	.513
	8.2	3.7×10^{-7}	2192	.534	2182	.542
	8.7	3.8×10^{-7}	2150	.630	2186	.596
	8.9	1.8×10^{-7}	1850	.633	1890	.591
	9.1	1.5×10^{-7}	1552	.680	1576	.649
	9.3	1.5×10^{-7}	1248	.728		
	9.6	1.5×10^{-7}	951	.734		
	9.7	1.5×10^{-7}	653	.777		



TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
COATING: ROKIDE MA (5MIL)
SUBSTRATE: COLUMBIUM–1% ZIRCONIUM
THERMOCOUPLE
RUN I - HEATING
RUN I - COOLING
OPTICAL PYROMETER
RUN I - HEATING
RUN I - COOLING
TOTAL HEMISPHERICAL EMITTANCE-ϵ_{TH}
0.9
0.8
0.7
0.6
0.5
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE — °F

Figure 16

SPECTRAL NORMAL EMITTANCE vs WAVE LENGTH

COATING: ROKIDE MA (5 MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM

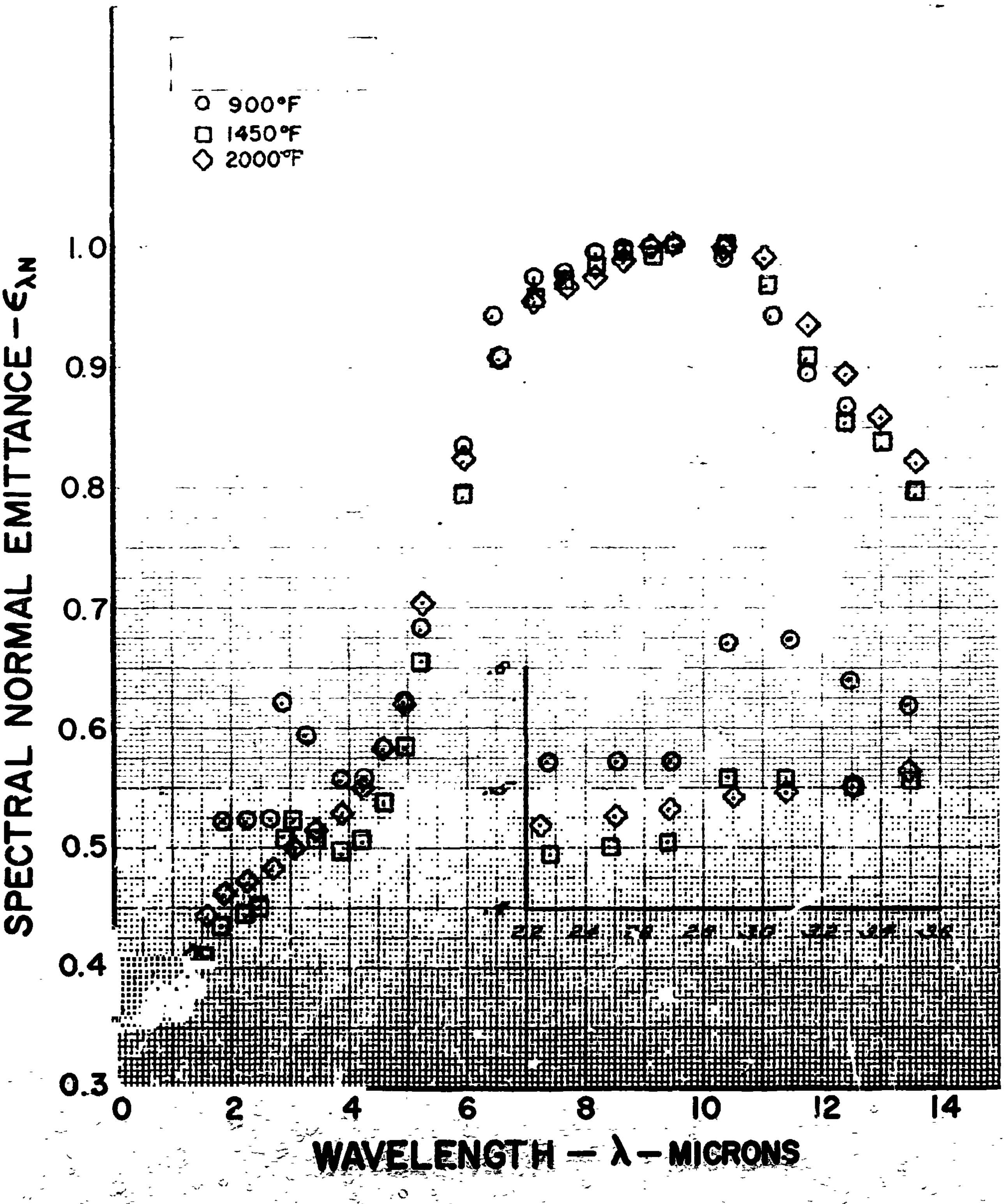


Figure 17

D. Chromic Oxide

Chromic oxide coatings were applied by two techniques, the Rokide process and by aluminum phosphate bonding. The typical analysis of Rokide C is as follows: 82.94 per cent Cr_2O_3, 8.39 per cent SiO_2, 3.16 per cent Al_2O_3, 2.96 per cent MgO, 1.28 per cent CaO, and 1.22 per cent (Fe_2O_3 + NaO_2 + TiO_2). Coatings of Rokide C were applied to columbium tubes and both spectral normal and total hemispherical emittance measurements were made. The aluminum phosphate bonded coating was on a columbium - 1 per cent zirconium tube and was tested in the total emittance rig only.

1. Rokide C Coatings

Before testing, the 3-mil thick coatings were blue-grey in color, extremely hard, and had a fine grit texture. They were very well bonded to the columbium substrate. Visual inspection of the specimens after testing showed that the coatings had changed from blue-grey to green.

a. **Total Hemispherical Emittance Test** - During the heating cycle total emittance increased from 0.72 at 400°F to 0.8 at 1000°F, remained constant to about 1600°F, and then decreased to about 0.7 at 2200°F. As the specimen was cooled, the emittance remained low, varying from 0.70 at 2200°F to 0.65 at 1500°F, indicating that a change in the coating had taken place.

As shown in Figure 18 and Table IX, the temperature indicated by the optical pyrometer and by the platinum-platinum 10 per cent rhodium thermocouple agreed very well at 1600°F and at 1800°F and then diverged. From this point on, it is assumed that the thermocouple data are unreliable. The emittance data, however, for the cooling portion of run 1 and for run 2, based on temperatures obtained with the thermocouple, are included in Figure 18 to show that the emittance dropped over the entire temperature range. Any changes in the coating that may have taken place during this test were not found by x-ray diffraction. The x-ray patterns showed the principal phase present to be Cr_2O_3 before and after the test.

TABLE IX

Coating: Rokide C
Substrate: Columbium
3.0 Mil Coating

Run No.	Elapsed Time (Hrs)	Pressure (mm Hg)	T. C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	1.1	3.3×10^{-6}	396	.717		
	2.2	2.1×10^{-6}	595	.767		
	2.6	2.1×10^{-6}	798	.778		
	3.0	3.3×10^{-6}	998.5	.811		
2	4.8	1.9×10^{-6}	1192.5	.798		
	5.3	1.9×10^{-6}	1405.5	.802		
	5.8	1.9×10^{-6}	1601.5	.807	1615	.786
	6.4	1.9×10^{-6}	1999	.793	2018	.767
	6.8	1.8×10^{-6}	1805.3	.790	1815	.777
	7.2	2.9×10^{-6}	2200.3	.751	2235	.702
	7.6	1.9×10^{-6}	2092	.722	2120	.682
	8.0	1.9×10^{-6}	1903	.701	1930	.669
	8.3	2.1×10^{-6}	1701	.686	1722	.660
	8.6	2.0×10^{-6}	1504	.679		
	8.8	2.0×10^{-6}	1302	.673		
3	9.2	2.3×10^{-6}	1351	.670		
	9.5	2.1×10^{-6}	1152.5	.664		
	10.0	1.9×10^{-6}	751	.657		
	10.4	1.9×10^{-6}	351	.642		
4	11.8	2.7×10^{-6}	398	.656		
	12.4	1.9×10^{-6}	604	.642		
	12.9	2.0×10^{-6}	800	.670		
	15.2	2.2×10^{-6}	979	.664		
5	15.6	2.1×10^{-6}	1100	.688		
	15.8	2.0×10^{-6}	1200	.667		
	16.1	2.0×10^{-6}	1298	.661		
	16.4	2.0×10^{-6}	1396	.687		
	16.7	2.0×10^{-6}	1500	.670	1523	.640

TABLE IX (Cont'd)

Run No.	Elapsed Time (Hrs)	Pressure (mm Hg)	T.C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
6	17.2	2.0×10^{-6}	1596	.686	1626	.647
	17.5	2.0×10^{-6}	1698	.680	1721	.651
	17.8	2.0×10^{-6}	1803	.686	1832	.652
	18.5	2.3×10^{-6}	1903	.702	1923	.679
	18.7	2.0×10^{-6}	2003	.709	2031	.678
	19.0	2.0×10^{-6}	2097	.724	2121	.697
	19.3	2.5×10^{-6}	2196	.759	2237	.714

TOTAL HEMISPHERICAL EMITTANCE VS TEMPERATURE

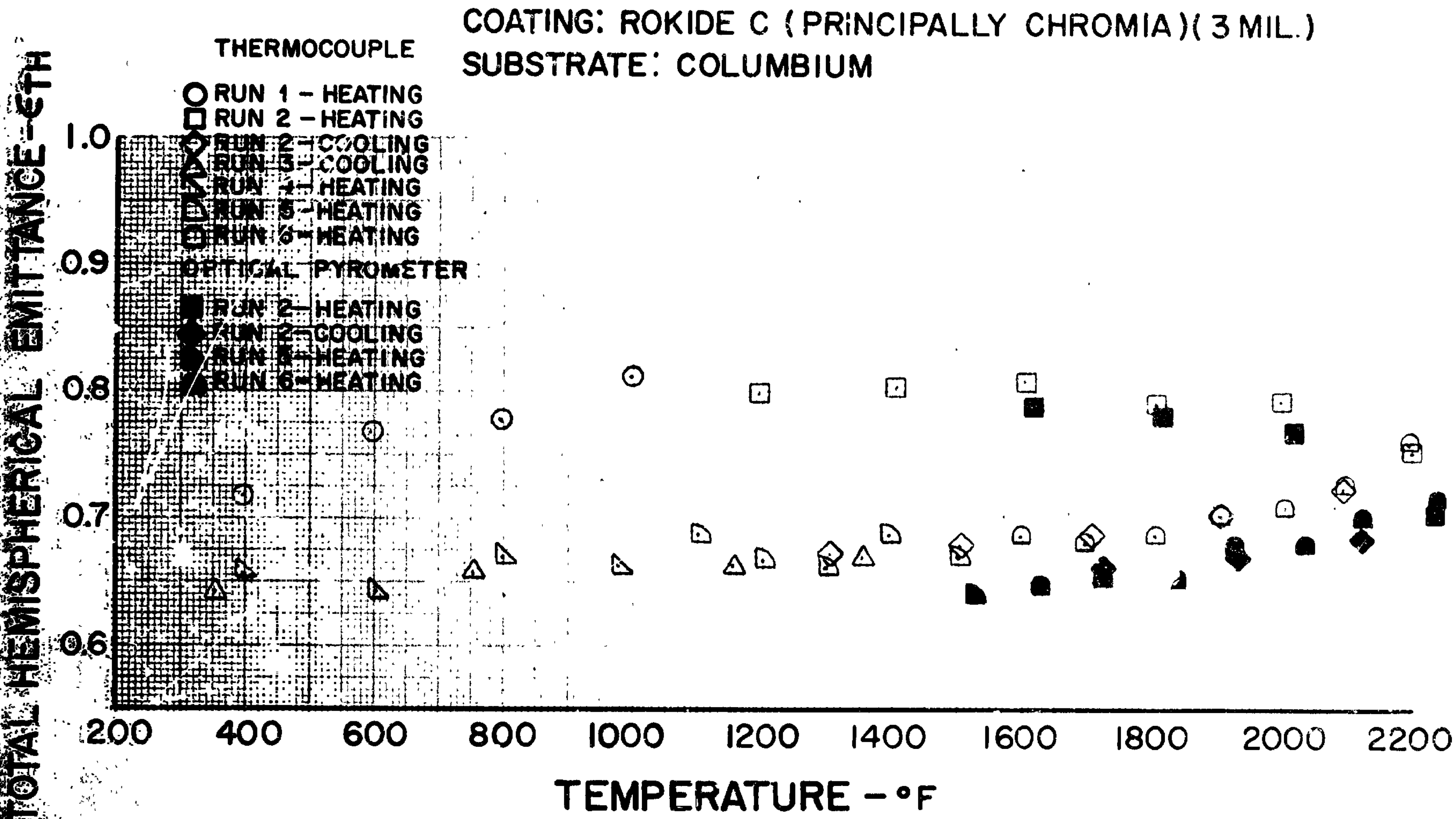


Figure 18

b. Spectral Normal Emittance Test - Spectral normal emittance was measured from 1.5 to 13.5 microns at 900°F, from 1 to 14 microns at 1450°F and from 1 to 14 microns at 2000°F. The data are shown in Figure 19. The emittances indicated are higher than had been anticipated from total emittance results discussed above and in PWA-2043. It is suspected that this difference is due to the quality of the black body being significantly less than unity. For this reason the black body hole configuration was changed from a rectangular slot (.028 in x .085 in) to two 0.023-inch diameter holes thus reducing the size of the black body to one-third of its original size.

2. Aluminum Phosphate Bonded Coating

The 3-mil thick coating was green in color and had a smooth (fine grit) surface. It was moderately hard and was well bonded to the substrate. Total emittance varied from 0.80 at 500°F to 0.73 at 1400°F. Beyond this point the behavior of the specimen was erratic and the data shown in Figure 20 is questionable at temperatures higher than 1400°F.

SPECTRAL NORMAL EMITTANCE VS WAVELENGTH

COATING: ROKIDE C (3 MIL)
SUBSTRATE: COLUMBIUM

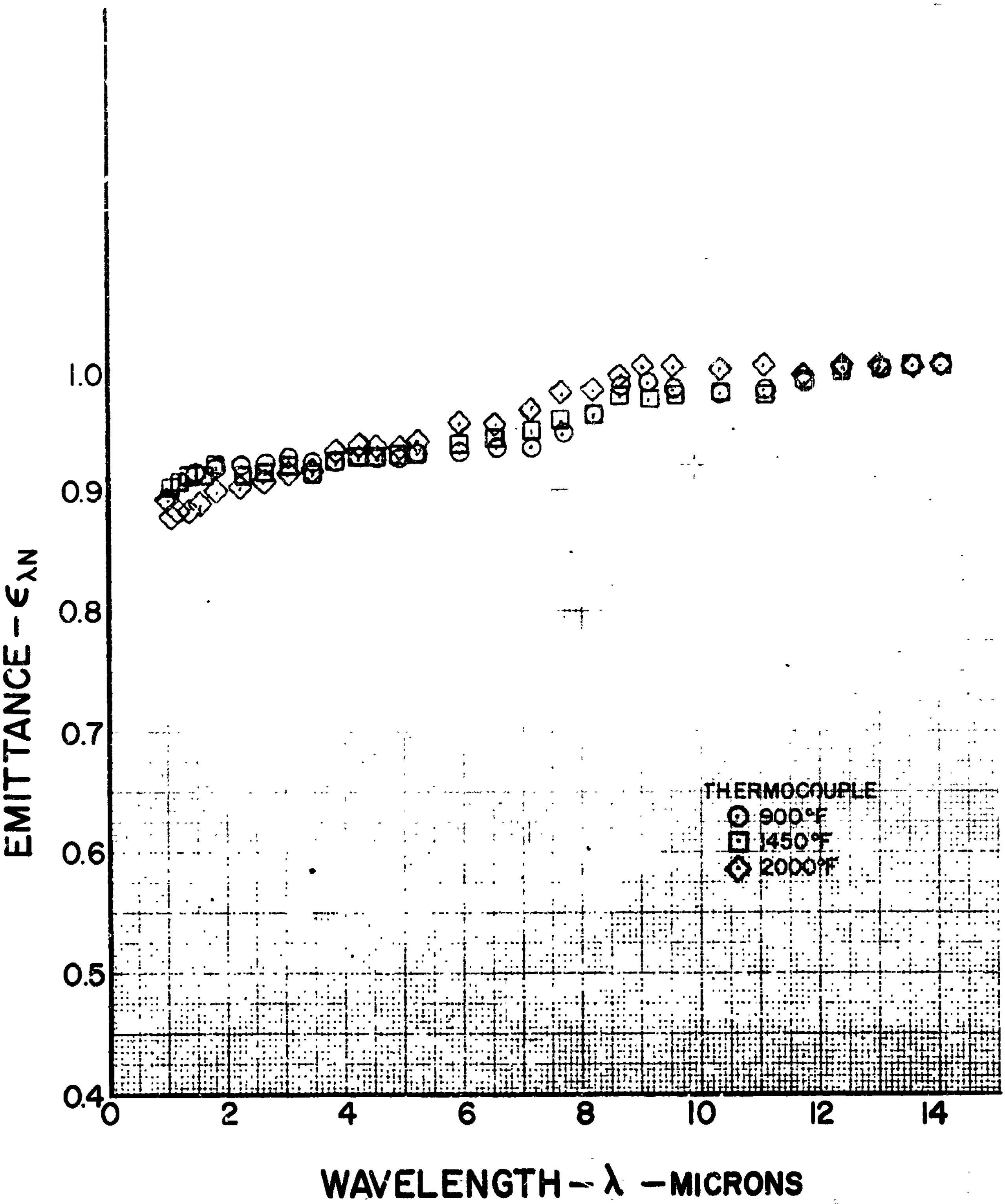


Figure 19

TABLE X

Coating: Chromia-Aluminum Phosphate Bonded
Substrate: Columbium - 1% Zirconium

3.0 Mil Coating

Run No.	Elapsed Time (Hrs)	Pressure (mm Hg)	T.C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	0.4	$2.2x10^{-7}$	305	.747		
	0.7	$2.1x10^{-7}$	497	.802		
	0.9	$2.4x10^{-7}$	698	.791		
	1.4	$2.6x10^{-7}$	904	.784		
	1.7	$7.1x10^{-7}$	1005	.769		
	1.9	$5.3x10^{-7}$	1101	.765		
	2.1	$1.2x10^{-6}$	1200	.758		
	2.3	$4.8x10^{-6}$	1301	.739		
	2.5	$2.0x10^{-6}$	1401	.727		
	2.8	$1.2x10^{-6}$			1512	.744
	3.4	$5.0x10^{-7}$			1655	.676
	3.8	$4.8x10^{-7}$			1820	.650

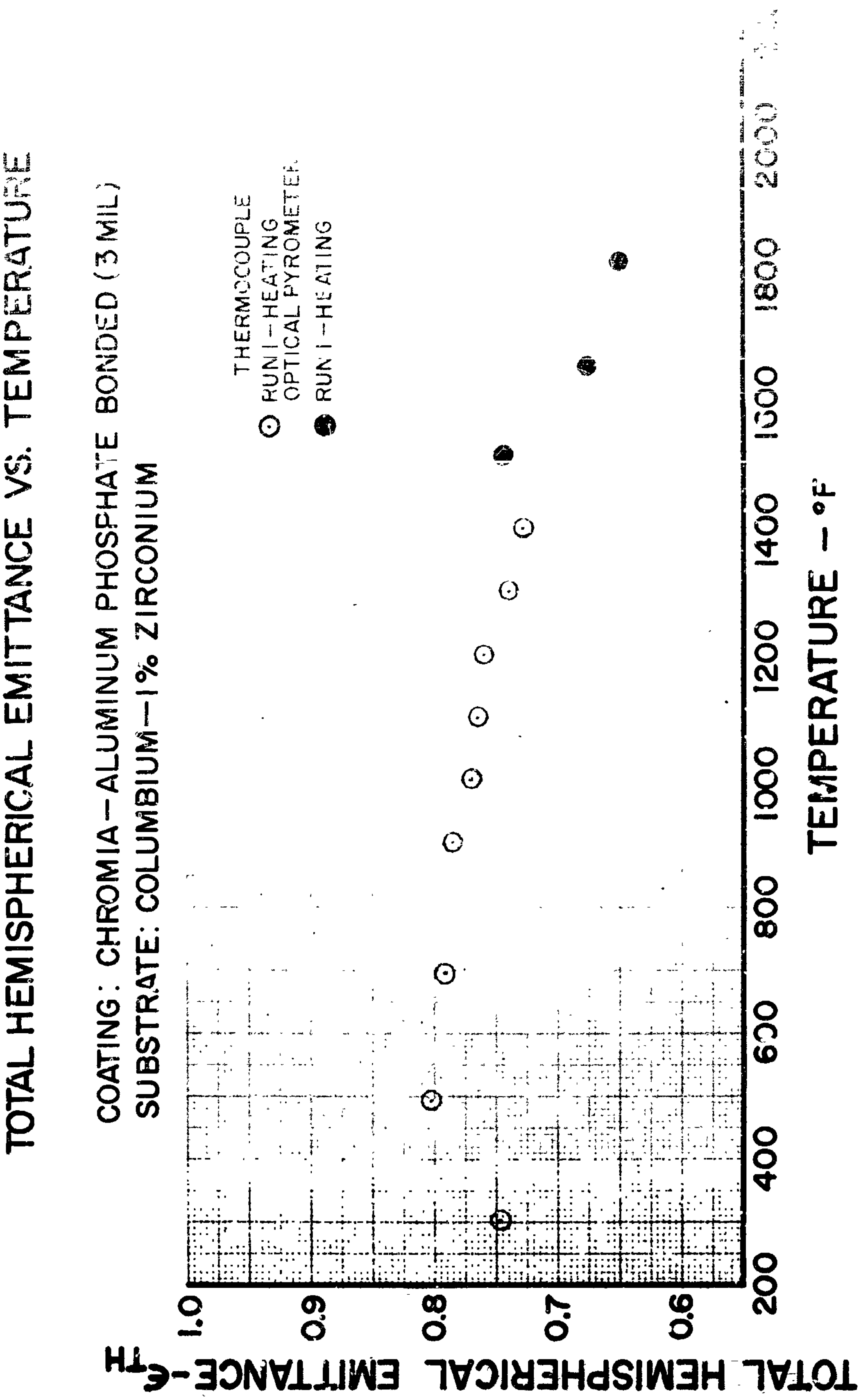

TOTAL HEMISPHERICAL EMITTANCE VS. TEMPERATURE
COATING: CHROMIA—ALUMINUM PHOSPHATE BONDED (3 MIL)
SUBSTRATE: COLUMBIUM—1% ZIRCONIUM
THERMOCOUPLE
RUN I - HEATING
OPTICAL PYROMETER
RUN I - HEATING
TOTAL HEMISPHERICAL EMITTANCE-ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE — °F

Figure 20

E. Ceric Oxide

A 2-mil coating of ceric oxide (CeO_2) supplied by the Variacoid Chemical Company was aluminum-phosphate bonded to a columbium - 1 per cent zirconium substrate and tested in the total emittance rig. Total emittance was measured from 300°F to 1700°F. As shown in Figure 21, total emittance varied from 0.75 at 300°F to 0.65 at 1300°F and then increased. Beyond 1300°F visual observations indicated fluctuations in the brightness of the surface. When the specimen was removed from the test rig it was noted that the color had changed from white to blue mottled with white. The low emittance level of this coating, at all temperatures, did not warrant further testing or material analysis.

TABLE XI

Coating: Ceria - Aluminum Phosphate Bonded
Substrate: Columbium - 1% Zirconium

2.0 Mil Coating

Run No.	Elapsed Time	Pressure (mm Hg)	T. C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	0.1	2.6×10^{-7}	301	.747		
	1.6	2.0×10^{-7}	699	.754		
	2.0	2.6×10^{-7}	901	.716		
	2.4	2.4×10^{-7}	1001	.680		
	2.7	3.1×10^{-7}	1100	.707		
	2.9	3.4×10^{-7}	1202	.660		
	3.1	4.0×10^{-7}	1301	.644		
	3.4	5.0×10^{-7}	1396	.660		
	4.1	5.0×10^{-7}	1495	.733	1470	.772
	5.0	4.0×10^{-7}	1599	.762	1608	.749
	5.3	3.2×10^{-7}	1698	.749	1735	.700

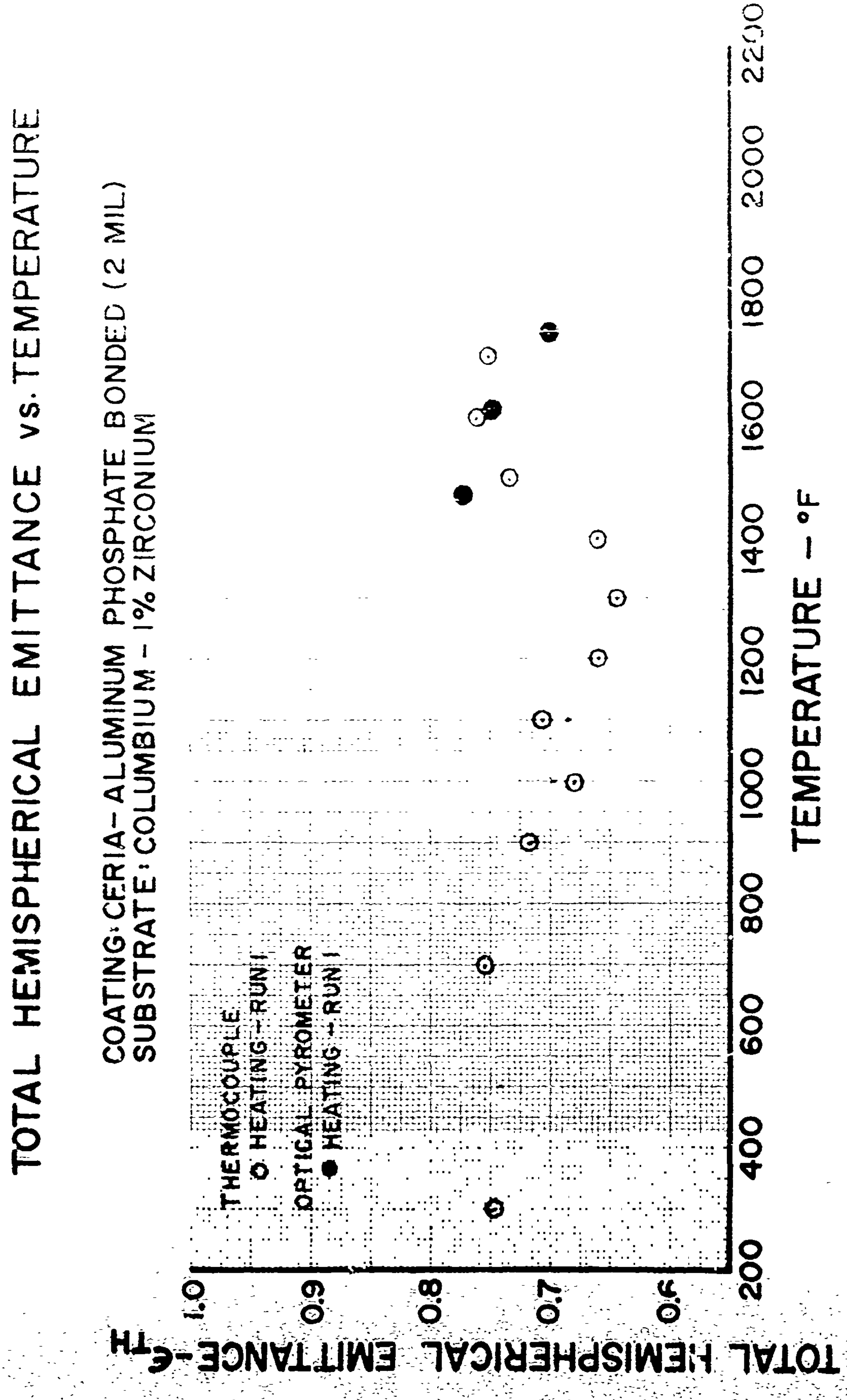

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
COATING: CERIA-ALUMINUM PHOSPHATE BONDED (2 MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
HEATING - RUN I
OPTICAL PYROMETER
HEATING - RUN I
TOTAL HEMISPHERICAL EMITTANCE - ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE — °F

Figure 21

F. Titania

Titanium dioxide (TiO_2) was plasma-arc sprayed onto columbium tubes by Pratt & Whitney Aircraft. The titania used was supplied by Metco, Inc. (XP-1114). Two specimens were prepared, one for testing in the total hemispherical emittance rig and one for testing in the spectral normal emittance rig. The coatings were blue-black, and hard. The coating - substrate bond strength was fair. Two specimens were tested, one in the total hemispherical emittance rig and one in the spectral normal emittance rig.

1. Total Hemispherical Emittance Test

The 3-mil thick coating was tested at temperatures ranging from 400°F to 2250°F. The emittance values (see Figure 22) varied from 0.75 at 400°F to 0.83 at 1400°F during the first two test runs. At higher temperatures the emittance decreased and continued to decrease with each successive run. A low value of 0.68 was recorded at 2250°F during the final run. Visible characteristics of the coating did not change during testing.

2. Spectral Normal Emittance Test

Spectral normal emittance of a 2.5-mil thick coating was measured from 1.5 to 13.5 microns at 900°F, from 1 to 13.5 microns at 1450°F, and from 0.9 to 13.5 microns at a nominal temperature of 2000°F. The temperature increased from 1980°F to 2125°F during the final test which indicates a decrease in total emittance. The spectral emittance data presented in Figure 23 shows evidence of this change. The 2000°F test was re-run with little change in temperature indicating that no further large changes in emittance were taking place. Spectral emittances were determined to be essentially the same at 900°F and 1450°F but were found to decrease drastically as the specimen was heated to 2000°F. This decrease in emittance is consistent with the similar decrease observed for the total hemispherical emittance specimen. The visible characteristics of the coating did not change as a result of testing.

Analysis of the coatings before and after testing by x-ray diffraction showed Ti_2O_3 as the principal phase present. Spectrographic analysis showed no change in the elements present.

TABLE XII
Coating: Titania-Plasma Arc Sprayed
Substrate. Columbium
3.0 Mil Coating

Run No.	Elapsed Time (Hrs)	Pressure (mm Hg)	T.C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	1.1	2.1×10^{-6}	386	.754		
	1.6	2.1×10^{-6}	601	.776		
	2.5	2.0×10^{-6}	799	.796		
	3.0	2.2×10^{-6}	1002.8	.809		
	3.6	2.0×10^{-6}	1200	.820		
	4.0	2.2×10^{-6}	1399.5	.825		
Heating Current Off; Vacuum Maintained						
2	4.5	3.0×10^{-6}	397.8	.767		
	5.1	2.3×10^{-6}	627.4	.770		
	5.6	2.4×10^{-6}	801.4	.799		
	6.4	2.4×10^{-6}	998.7	.809		
	6.8	2.6×10^{-6}	1201.7	.845		
	7.2	2.75×10^{-6}	1399.7	.833	1400	.832
	7.6	2.5×10^{-6}	1596.1	.847	1605	.832
	8.0	2.4×10^{-6}			1825	.807
3	9.2	2.3×10^{-6}			1605	.810
	9.8	3.4×10^{-6}			1810	.799
	10.3	8.0×10^{-6}			2025	.791
Heating Current Off; Rig Opened						
4	10.9	2.2×10^{-6}			1608	.764
	11.6	2.1×10^{-6}			1800	.751
	11.9	2.2×10^{-6}			2012	.751
	12.3	2.3×10^{-6}			2250	.680

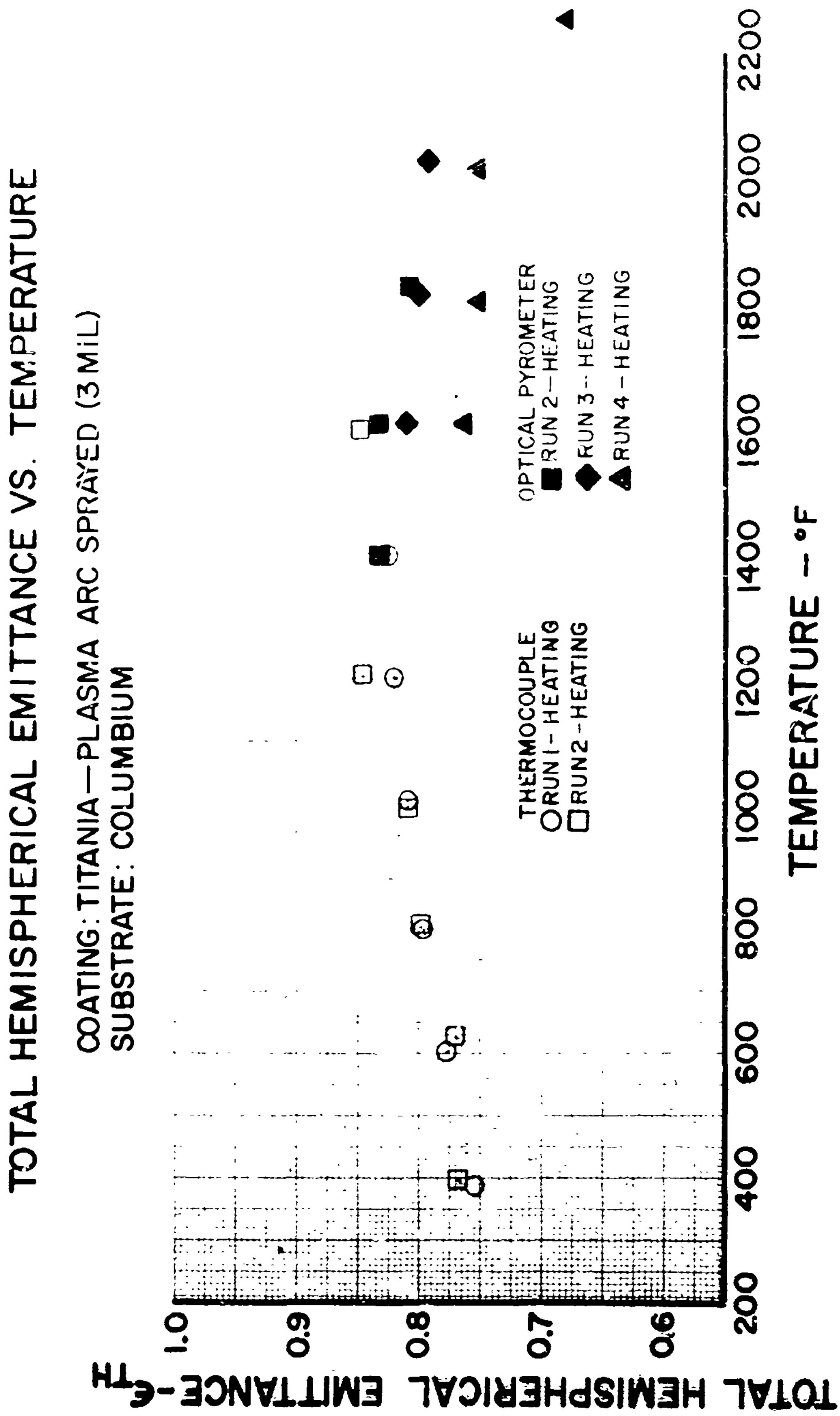

TOTAL HEMISPHERICAL EMITTANCE VS. TEMPERATURE
COATING: TITANIA—PLASMA ARC SPRAYED (3 MIL)
SUBSTRATE: COLUMBIUM
THERMOCOUPLE
RUN 1 - HEATING
RUN 2 - HEATING
OPTICAL PYROMETER
RUN 2 - HEATING
RUN 3 - HEATING
RUN 4 - HEATING
TEMPERATURE — °F
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TOTAL HEMISPHERICAL EMITTANCE - ϵ_{TH}
1.0
0.9
0.8
0.7
0.6

Figure 22

SPECTRAL NORMAL EMITTANCE VS WAVELENGTH

COATING: TITANIA— PLASMA ARC SPRAYED (2.5 MIL)
SUBSTRATE: COLUMBIUM

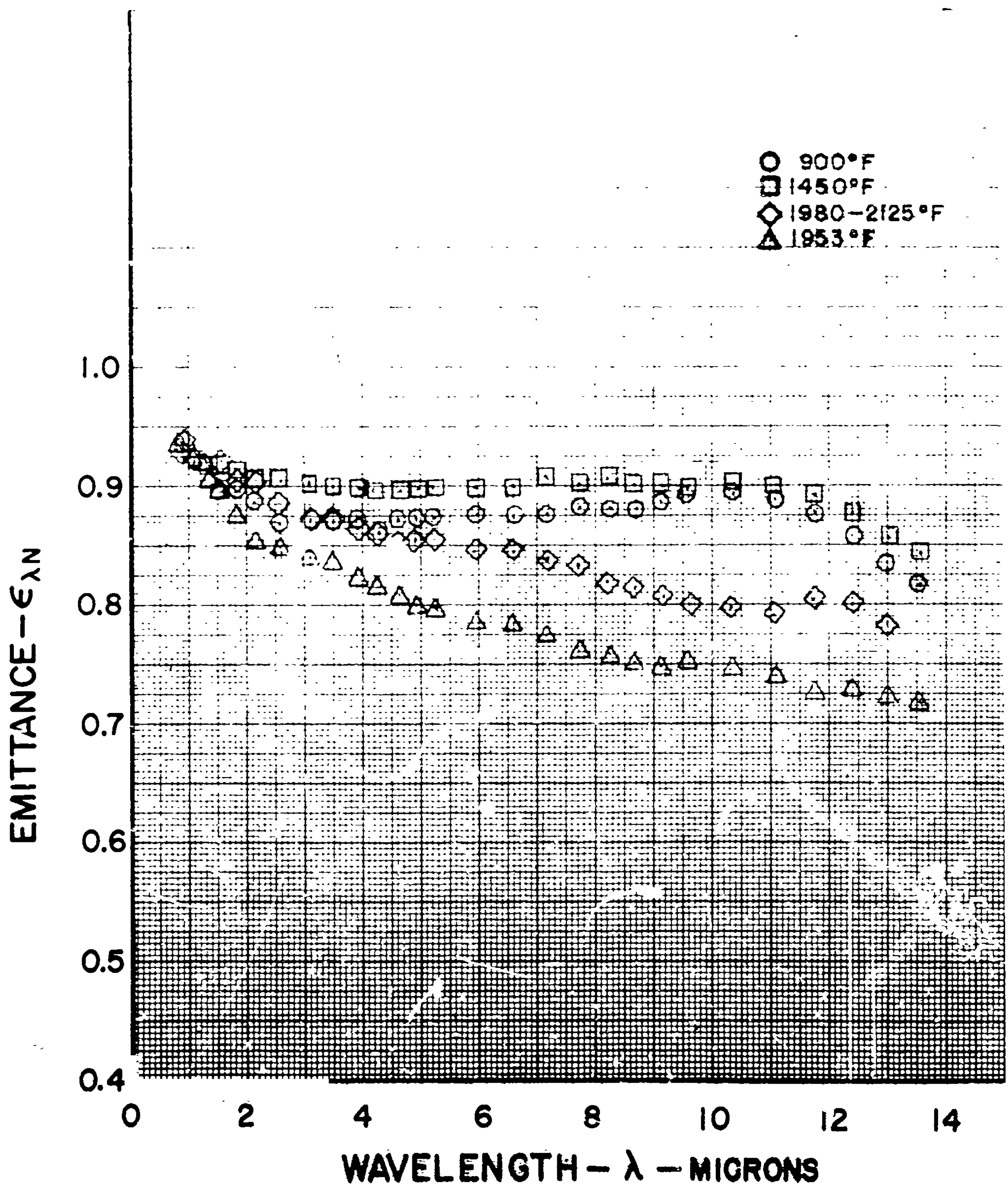


Figure 23

C. Barium Titanate

Barium titanate was plasma-arc-sprayed onto columbium-1 per cent zirconium tubes. The barium-titanate powder was obtained from Continental Coatings Corporation (FCE-11). Two specimens were prepared at the same time, one for testing in the total hemispherical emittance rig, and the other for testing in the spectral normal emittance rig.

1. Total Hemispherical Emittance Test

The 5-mil thick coating was grey-black, had a fine grit texture, and was well bonded to the substrate. Emittance values varied from 0.75 at 300°F to about 0.84 at 1600°F, as shown in Figure 24. At higher temperatures the emittance dropped to a low value of 0.64 at 2250°F. As the specimen was cooled, it was observed that the emittance had dropped over the entire temperature range indicating that a change in the coating had occurred. The color of the coating had changed to black mottled with violet.

2. Spectral Normal Emittance Test

The 5-mil thick coating was grey-black and hard. It had a fine grit texture and was well bonded to the substrate. Measurements were made at 900°F, 1450°F and 2000°F.

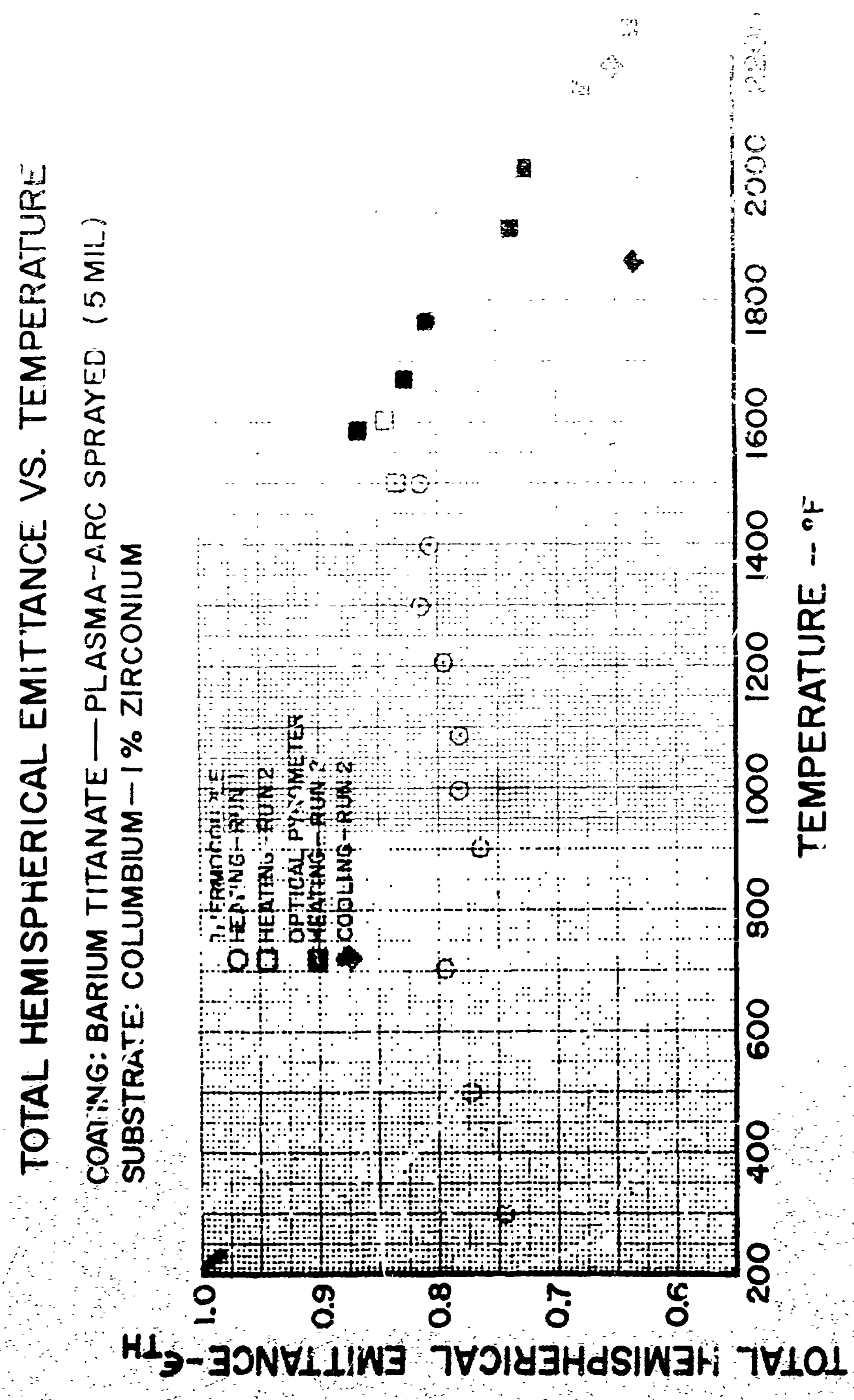
Since the shapes of the spectral emittance curves are quite different, (see Figure 25) it is assumed that a change in the coating took place. This is consistent with the results of the total emittance test described above. A change in color was also observed but the final color was brown rather than the violet and black of the total emittance specimen.

X-ray diffraction and spectrographic analysis showed no change in the coating as a result of testing. Both before and after testing the primary phase present was $BaO \cdot TiO_2$.

TABLE XIII

Coating : Barium Titanate - Plasma Arc Sprayed
Substrate : Columbium - 1% Zirconium
5 Mil Coating

Run No.	Elapsed Time (Hrs)	Pressure (mm H_g)	T.C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	0.7	6.9×10^{-7}	303	.745		
	1.1	5.1×10^{-7}	501	.772		
	1.4	5.4×10^{-7}	701	.794		
	1.8	6.5×10^{-7}	902	.765		
	2.1	7.3×10^{-7}	998	.783		
	2.3	6.0×10^{-7}	1084	.781		
	2.5	1.7×10^{-6}	1206	.794		
	2.7	5.2×10^{-7}	1300	.815		
	2.8	5.3×10^{-7}	1399	.806		
	2.9	5.8×10^{-7}	1501	.813		
Heating Current Off; Vacuum Maintained						
2	3.5	4.5×10^{-7}	1502	.831		
	4.1	4.6×10^{-7}	1606	.832	1586	.865
	4.6	5.0×10^{-7}			1668	.827
	5.0	4.8×10^{-7}			1768	.811
	5.7	6.7×10^{-7}			1920	.738
	6.0	6.7×10^{-7}			2015	.727
	6.3	9.0×10^{-7}			2150	.676
	6.6	9.0×10^{-7}			2244	.636
	6.9	6.2×10^{-7}			2184	.651
	7.6	3.1×10^{-7}			1860	.635
	7.8	3.7×10^{-7}			1546	.653



TOTAL HEMISPHERICAL EMITTANCE VS. TEMPERATURE
COATING: BARIUM TITANATE — PLASMA-ARC SPRAYED (5 MIL)
SUBSTRATE: COLUMBIUM — 1% ZIRCONIUM
THERMOCOUPLE
HEATING—RUN 1
HEATING—RUN 2
OPTICAL PYROMETER
HEATING—RUN 2
COOLING—RUN 2
TOTAL HEMISPHERICAL EMITTANCE-ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE — °F

Figure 24

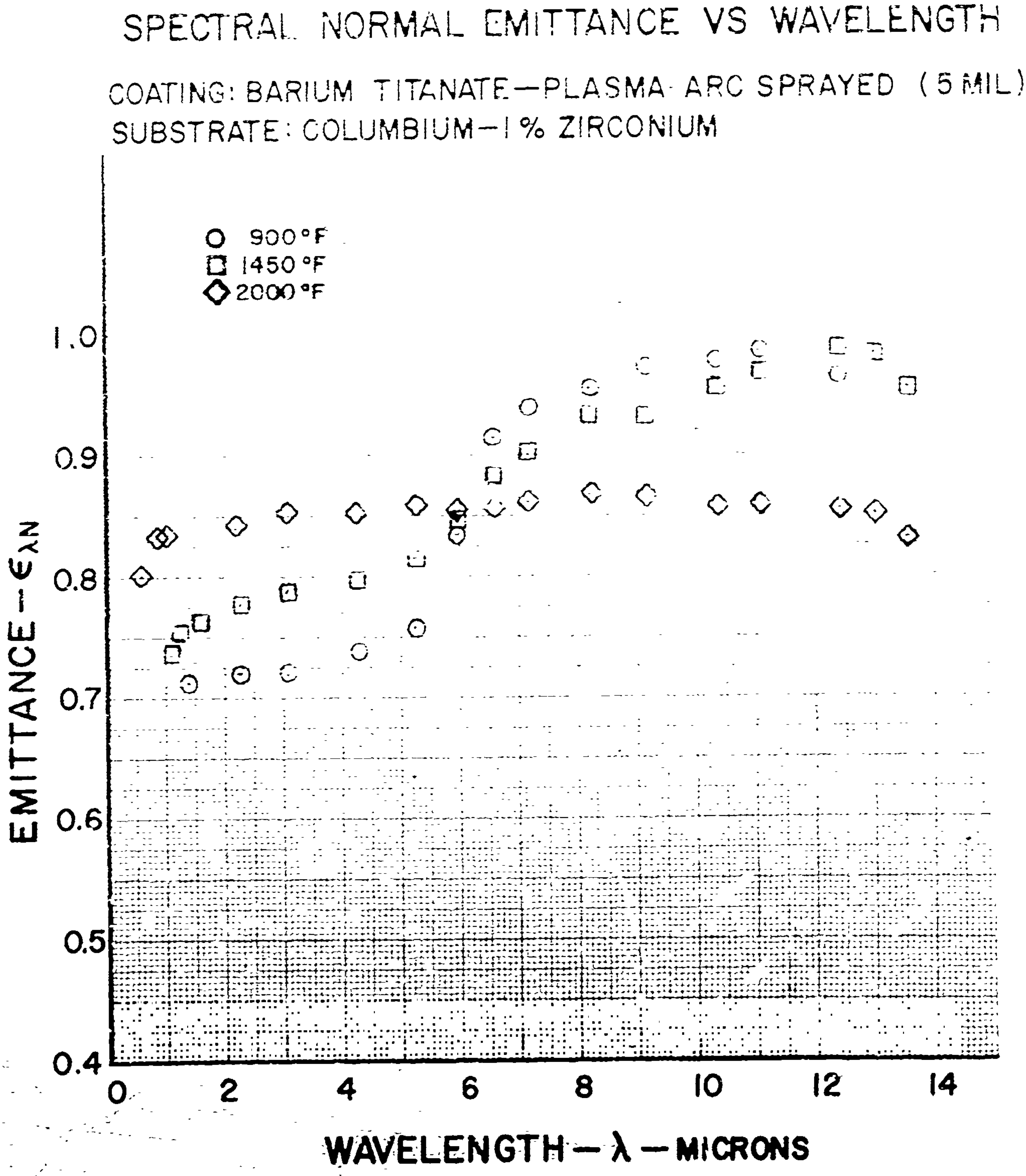

SPECTRAL NORMAL EMITTANCE VS WAVELENGTH
COATING: BARIUM TITANATE—PLASMA ARC SPRAYED (5 MIL)
SUBSTRATE: COLUMBIUM—1 % ZIRCONIUM
900 °F
1450 °F
2000 °F
EMITTANCE — $\epsilon_{\lambda N}$
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0
2
4
6
8
10
12
14
WAVELENGTH — λ — MICRONS

Figure 25

H. Calcium Titanate

Several specimens were prepared by plasma-arc spraying and by aluminum phosphate bonding calcium titanate powder, obtained from Metco, Inc., to columbium and columbium-1 per cent zirconium substrates. The powder used throughout this test, as well as that used to prepare the endurance specimen reported in PWA-2088, was from the same batch.

1. Total Hemispherical Emittance Test

Both types of coatings were tested in the total emittance rig and a comparison of the results indicates the differences in emittance characteristics that can occur by using two different coating methods.

a. Plasma Arc-Sprayed Coating - The plasma arc-sprayed coating was 5 mils thick, extremely hard, grey-black in color, had a rough texture, and was very well bonded to the columbium substrate.

As shown in Figure 26, the total emittance of the plasma arc-sprayed coating varied from 0.74 at 400°F to 0.85 at 1500°F during the initial heating, and then dropped to 0.74 at 2200°F. The drop in emittance at the high temperature appears to be the result of a permanent change since the level of emittance was lower during cooling over the entire temperature range. The increase in emittance during the first portion of the test may be a result of a change in the coating. Similar increases were noted in other tests with plasma arc-sprayed coatings of calcium titanate, strontium titanate and barium titanate. However, the emittance of this specimen was slightly lower than has previously been reported in PWA-2012 for the same plasma arc-sprayed coating. No visible changes in the coating were observed as the result of testing.

b. Aluminum Phosphate Bonded Coating - The aluminum phosphate bonded coating was applied to a columbium-1 per cent zirconium substrate producing a 5-mil thick coating that was hard on the surface and soft underneath, probably due to insufficient curing time. The coating was cream colored with a medium grit texture and was well bonded to the substrate.

TABLE XIV

Coating: Calcium Titanate - Plasma Arc Sprayed
Substrate: Columbium

5 Mil Coating

Run No.	Elapsed Time (Hrs.)	Pressure (mm Hg)	T.C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	1.2	2.1×10^{-6}	403	.732		
	1.5	2.1×10^{-6}	603	.768		
	1.9	2.0×10^{-9}	800	.796		
	2.2	2.4×10^{-6}	999	.810		
	2.4	2.0×10^{-6}	1104	.823		
	2.7	2.2×10^{-6}	1204	.817		
	4.1	2.0×10^{-6}	1307	.834		
	4.3	2.0×10^{-6}	1403	.839		
	4.6	2.2×10^{-6}	1502	.844	1500	.848
	5.1	2.6×10^{-6}	1601	.846	1605	.840
	5.7	2.4×10^{-6}	1701	.850	1710	.836
	6.2	2.0×10^{-6}	1801	.828	1811	.813
	6.5	2.1×10^{-6}	1900	.810	1912	.795
	6.8	2.6×10^{-6}	2004	.800	2018	.782
	7.1	2.9×10^{-6}	2105	.731	2111	.724
	7.3	3.6×10^{-6}	2197	.738	2198	.738
	7.4	2.8×10^{-6}	2150	.729	2166	.712
	7.7	2.1×10^{-6}	1950	.732	1967	.711
2	8.0	2.4×10^{-6}	1952	.788	1967	.769
	8.3	2.0×10^{-6}	1651	.797	1660	.784
	8.5	2.0×10^{-6}	1352	.784		
	8.7	2.1×10^{-6}	1150	.784		
	9.0	2.2×10^{-6}	744	.769		
	9.2	2.2×10^{-6}	352	.708		

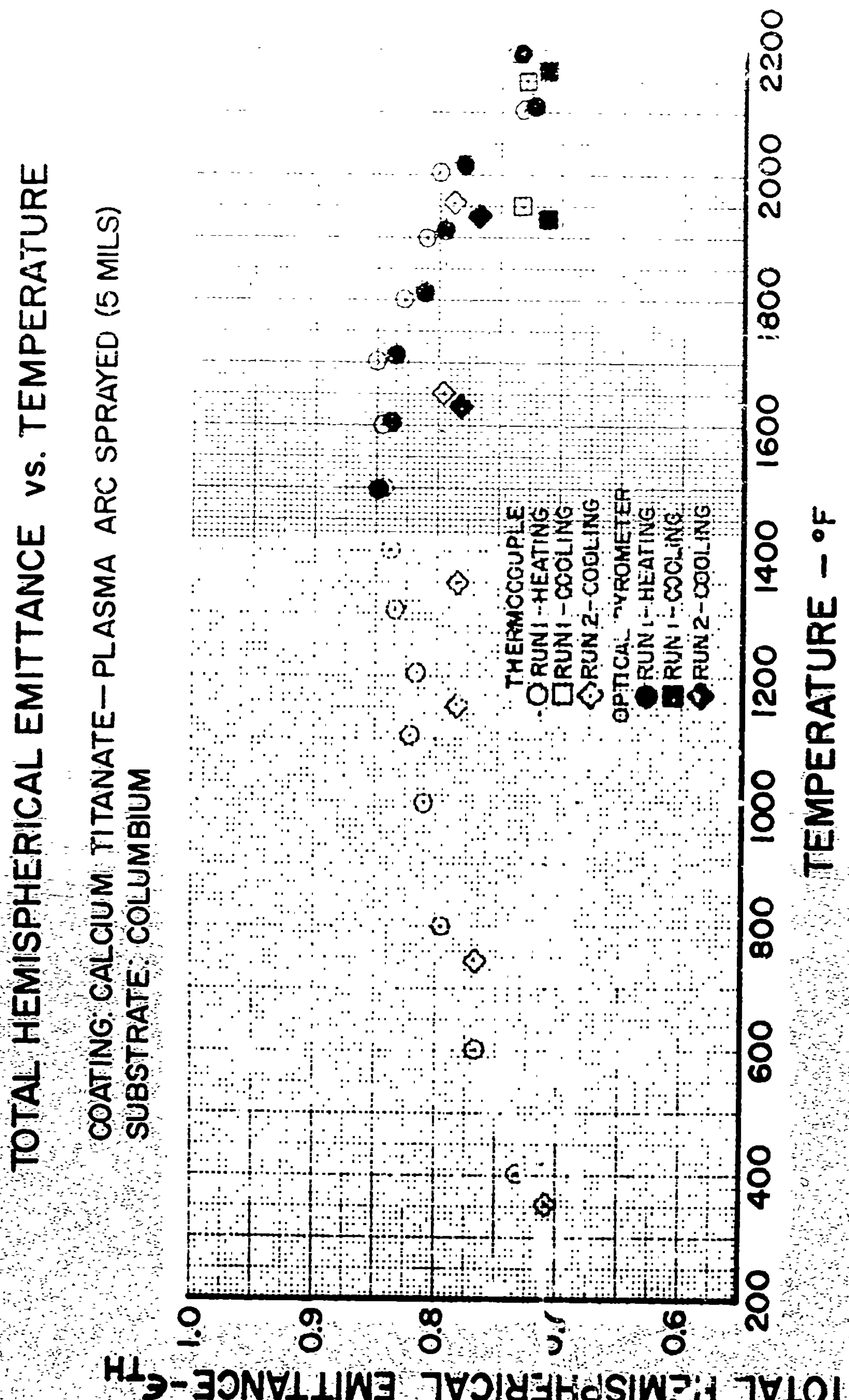

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
COATING: CALCIUM TITANATE—PLASMA ARC SPRAYED (5 MILS)
SUBSTRATE: COLUMBIUM
THERMOCOUPLE
RUN 1 -HEATING
RUN 1 -COOLING
RUN 2 -COOLING
OPTICAL PYROMETER
RUN 1 -HEATING
RUN 1 -COOLING
RUN 2 -COOLING
TEMPERATURE — °F
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
1.0
0.9
0.8
0.6
TOTAL HEMISPHERICAL EMITTANCE-ϵTH

Figure 26

Total emittance was measured from 300 to 1400°F. As shown in Figure 27, the total hemispherical emittance decreased from 0.88 at 300°F to 0.61 at 1100°F and then remained relatively constant up to 1400°F. The coating failed when heated above 1400°F and had changed to a white color during testing.

Calcium titanate, when applied by this method, may be of interest for low temperature radiators, i.e., for use at temperatures lower than 500°F. The coating, as indicated by its white color, probably has a low solar absorptance.

2. Spectral Normal Emittance Test

a. Plasma Arc-Sprayed Coating - The 3.5-mil thick coating, plasma arc-sprayed onto a columbium substrate, was grey-black, extremely hard, had a rough texture, and was very well bonded to the substrate. Spectral emittance was measured at 900°F, 1450°F and 2000°F. The spectral emittance data is presented in Figure 28. It may be noted that the general level of the spectral emittance curve is highest at 1450°F. This is consistent with the changes noted above in the discussion of the total emittance test of the same coating. That is, an increase in emittance took place at some temperature below 1500°F and when the specimen was heated to 2000°F a decrease in emittance took place. The data reported in PWA-2012 shows the rise in emittance that occurred below 1500°F to be a permanent change for temperatures up to about 1675°F. However, as shown in Figure 26, heating the specimen to temperatures above 1700°F causes the emittance to decrease. This decrease appears to be a result of a second permanent change. This second change is consistent with the decrease in the level of the spectral emittance curve at 2000°F. The specimen showed no change in visible characteristics as a result of testing.

b. Aluminum Phosphate-Bonded Coating - The 5-mil thick coating was hard, cream colored, had a medium grit texture, and before testing had a good bond to the columbium-1 per cent zirconium substrate. When this specimen was heated above 900°F, however, cracks developed in the coating resulting in

TABLE XV

Coating: Calcium Titanate-Aluminum Phosphate Bonded
Substrate: Columbium - 1% Zirconium

5.0 Mil Coating

Run No.	Elapsed Time (Hrs)	Pressure (mm Hg)	T.C. (°F)	ϵ_{TH}
1	3.3	1.7×10^{-7}	301	.883
	4.1	1.6×10^{-7}	496	.838
	4.7	1.8×10^{-7}	699	.793
	5.3	5.0×10^{-7}	901	.687
	5.7	5.3×10^{-7}	1005	.634
	6.0	3.8×10^{-7}	1012	.621
	6.2	4.4×10^{-7}	1099	.608
	6.5	3.6×10^{-7}	1196	.607
	6.7	3.8×10^{-7}	1297	.616
	7.2	2.5×10^{-7}	1386	.605

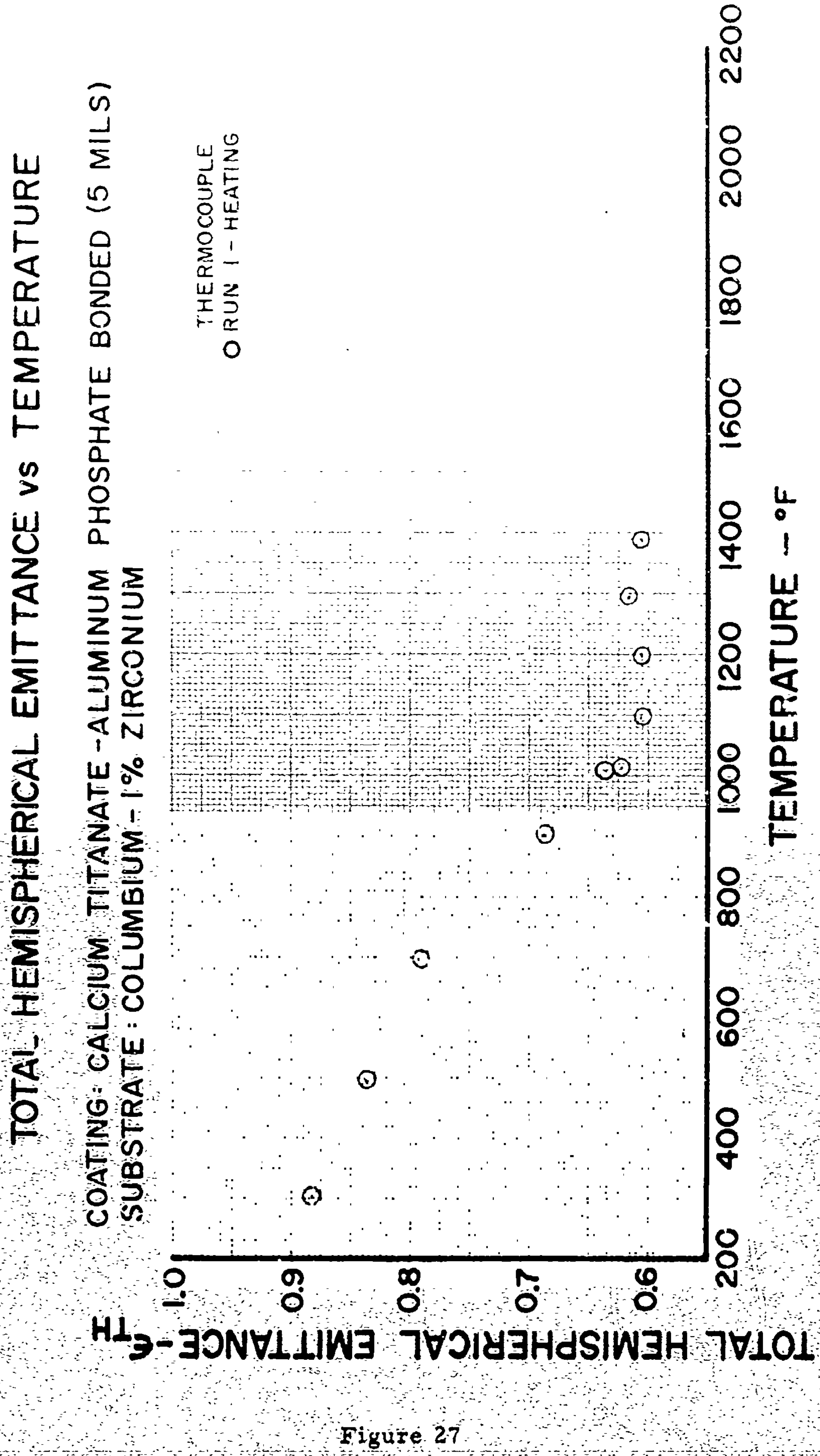

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: CALCIUM TITANATE -ALUMINUM PHOSPHATE BONDED (5 MILS)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
O RUN 1 - HEATING
TOTAL HEMISPHERICAL EMITTANCE - ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE — °F

Figure 27

SPECTRAL NORMAL EMITTANCE vs WAVELENGTH

COATING: CALCIUM TITANATE—PLASMA ARC SPRAYED (3.5 MILS)
SUBSTRATE: COLUMBIUM

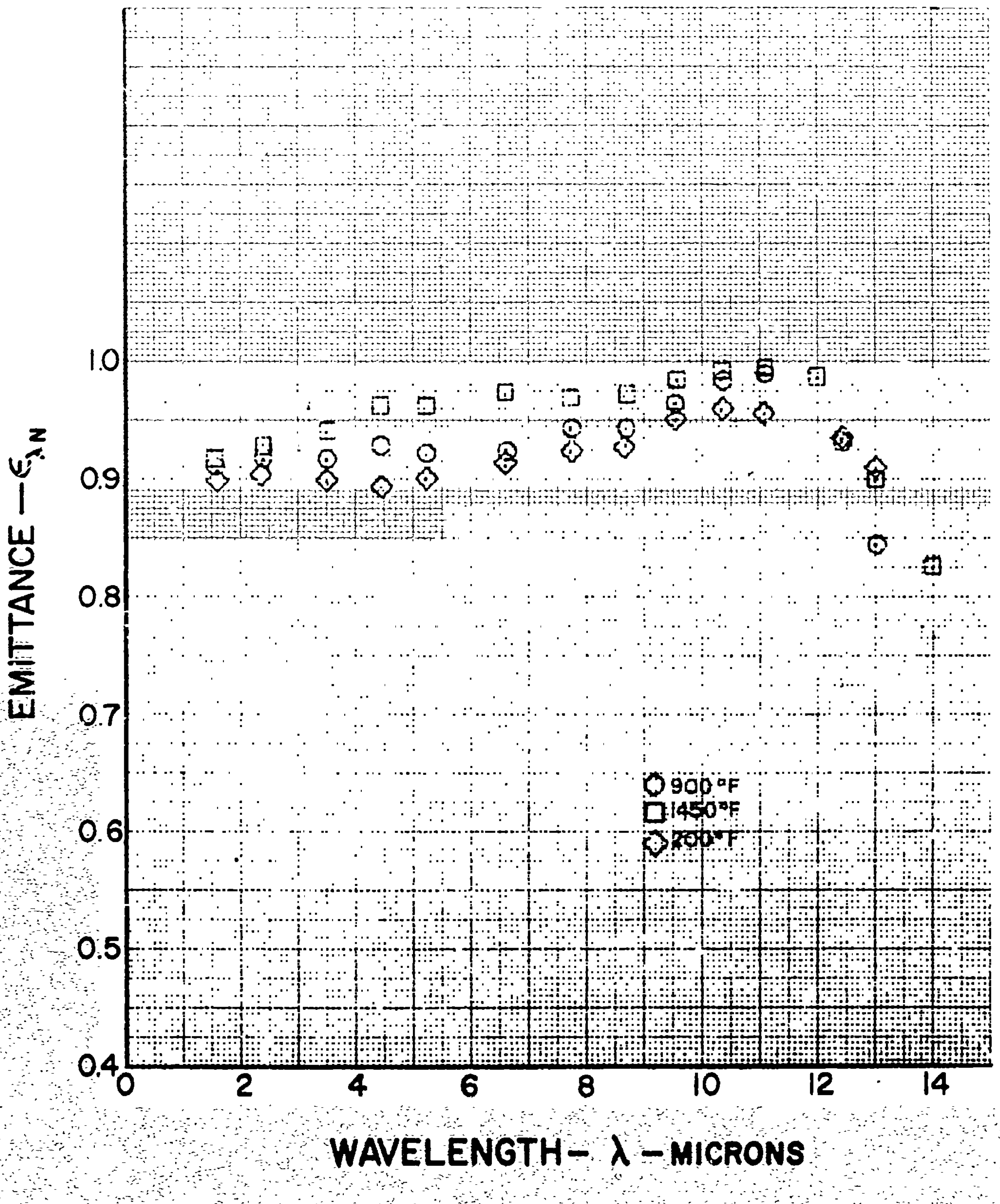


Figure 28

unreliable data. After testing the coating was white with blue blotches. Test results are shown in Figure 29.

3. Calcium Titanate - General Remarks

X-ray diffraction patterns were obtained for each of the specimens tested and these showed no change in the structure of any of the specimens as a result of testing. The principle phase present in each case was $CaO \cdot TiO_2$. Spectrographic analysis was performed on the aluminum phosphate-bonded coating tested in the spectral emittance rig and this showed no change in the elements present as a result of testing.

SPECTRAL NORMAL EMITTANCE vs WAVELENGTH

COATING: CALCIUM TITANATE-ALUMINUM PHOSPHATE BONDED (5 MILS)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM

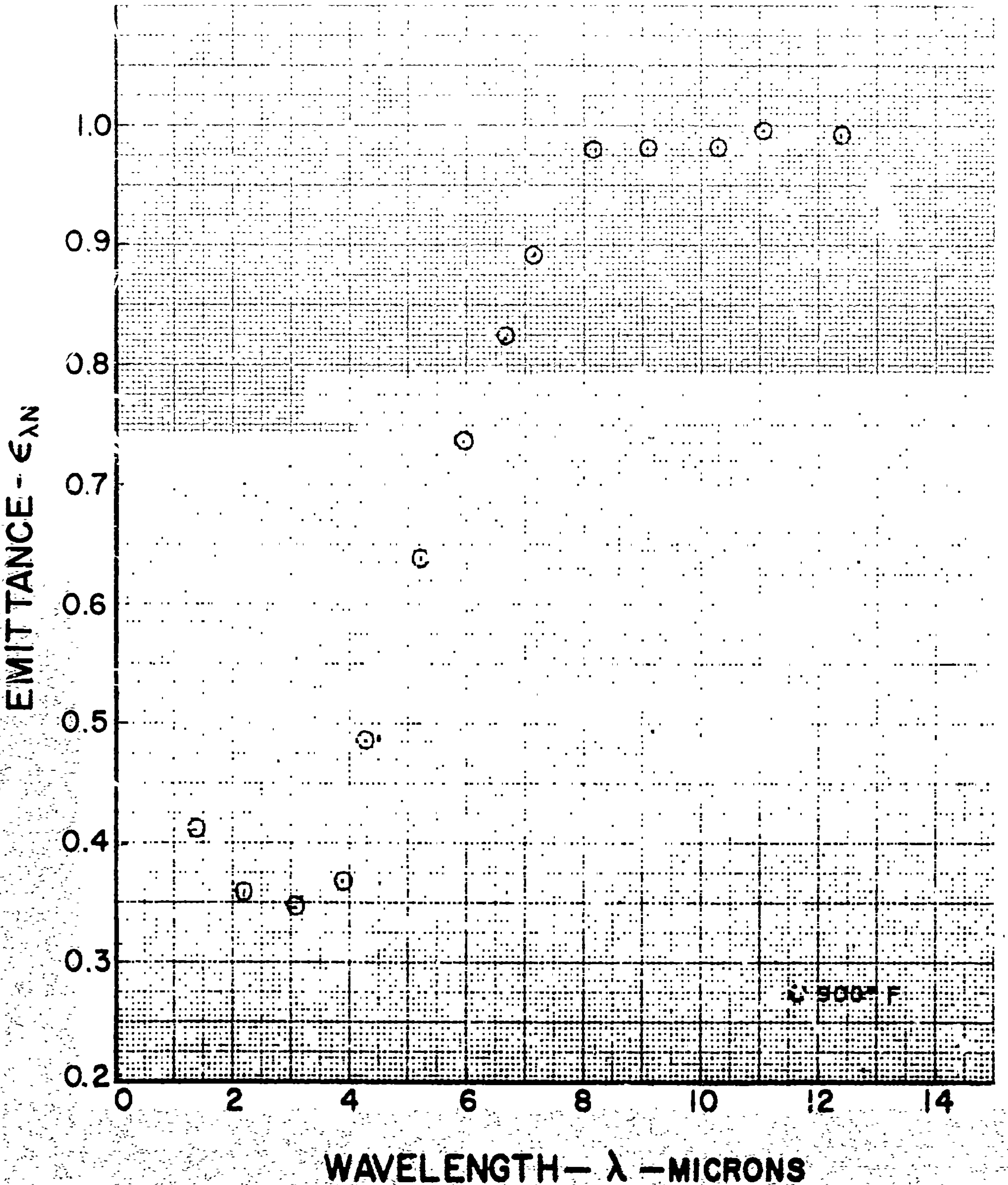


Figure 29

I. Strontium Titanate

Several coatings were prepared by plasma-arc spraying strontium titanate powder, obtained from the Plasmadyne Corporation, orto columbium-1 per cent zirconium tubes. Two separate sets of specimens were prepared using powder from the same container for both sets. The specimens were tested in the total hemispherical and spectral normal emittance rigs.

1. Total Hemispherical Emitance Test

One specimen from each of the two batches was tested in the total hemispherical emittance rig because the quality of the coating in the first batch was not as good as was desired.

a. First Specimen - The first coating tested was 2 mils thick, light grey in color, moderately hard, had a smooth texture and was fairly well bonded to the substrate. This specimen was tested over the temperature range of 200 to 2100°F. As may be seen in Figure 30, the total hemispherical emittance remained relatively constant at about 0.68 from 200°F to 700°F and then increased to about 0.88 at 1400°F where it remained constant up to about 1600°F. As the specimen was heated above 1600°F the emittance decreased to about 0.5 at 2125°F. During cooling the emittance remained at this low level, indicating that the drop was due to a change in the coating. The increase in emittance between 700° and 1400°F is also believed to be a result of a change in the coating material. This phenomenon was observed in an earlier test (reported in PWA-2043) in which a similar increase was noted. The specimen in that test was reinstrumented with new thermocouples and emittance was measured a second time. The values obtained in this latter test run indicated that the emittance had increased over the entire temperature range of 300° to 1450°F.

Figure 30 indicates that between 1700°F and 2000°F the total emittance data based on optical pyrometer temperature measurements do not agree very well with those based on thermocouple measurements. This is somewhat misleading because the temperature was steadily increasing during this period although the electrical power input to the specimen

TABLE XVI

Coating: Strontium Titanate - Plasma Arc Sprayed
Substrate: Columbium - 1% Zirconium

2 Mil Coating

Run No.	Elapsed Time (Hrs.)	Pressure (mm Hg)	T.C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	0.9	2.5×10^{-6}	218	.690		
	1.2	2.3×10^{-6}	304	.694		
	1.7	2.2×10^{-6}	493	.693		
	2.1	2.2×10^{-6}	686	.683		
	2.5	2.2×10^{-6}	897	.708		
	3.0	2.3×10^{-6}	992	.743		
Heating Current Off; Vacuum Maintained						
2	3.5	2.4×10^{-6}	1099	.789		
	3.7	2.5×10^{-6}	1198	.832		
	3.9	2.4×10^{-6}	1300	.862		
	4.1	2.4×10^{-6}	1405	.875		
	4.3	2.7×10^{-6}	1506	.875	1498	.880
	4.6	2.4×10^{-6}	1603	.873	1593	.890
	5.0	2.5×10^{-6}	1713	.859	1700	.880
	5.2	2.9×10^{-6}	1813	.834	1830	.810
	5.6	2.9×10^{-6}	1933	.708	1969	.667
	6.0	2.6×10^{-6}	20[illegible]9	.610	2030	.599
	7.1	2.2×10^{-6}			2128	.507
	7.3	2.0×10^{-6}			2000	.493
	7.7	2.0×10^{-6}			1774	.468
	8.0	1.9×10^{-6}			1418	.535

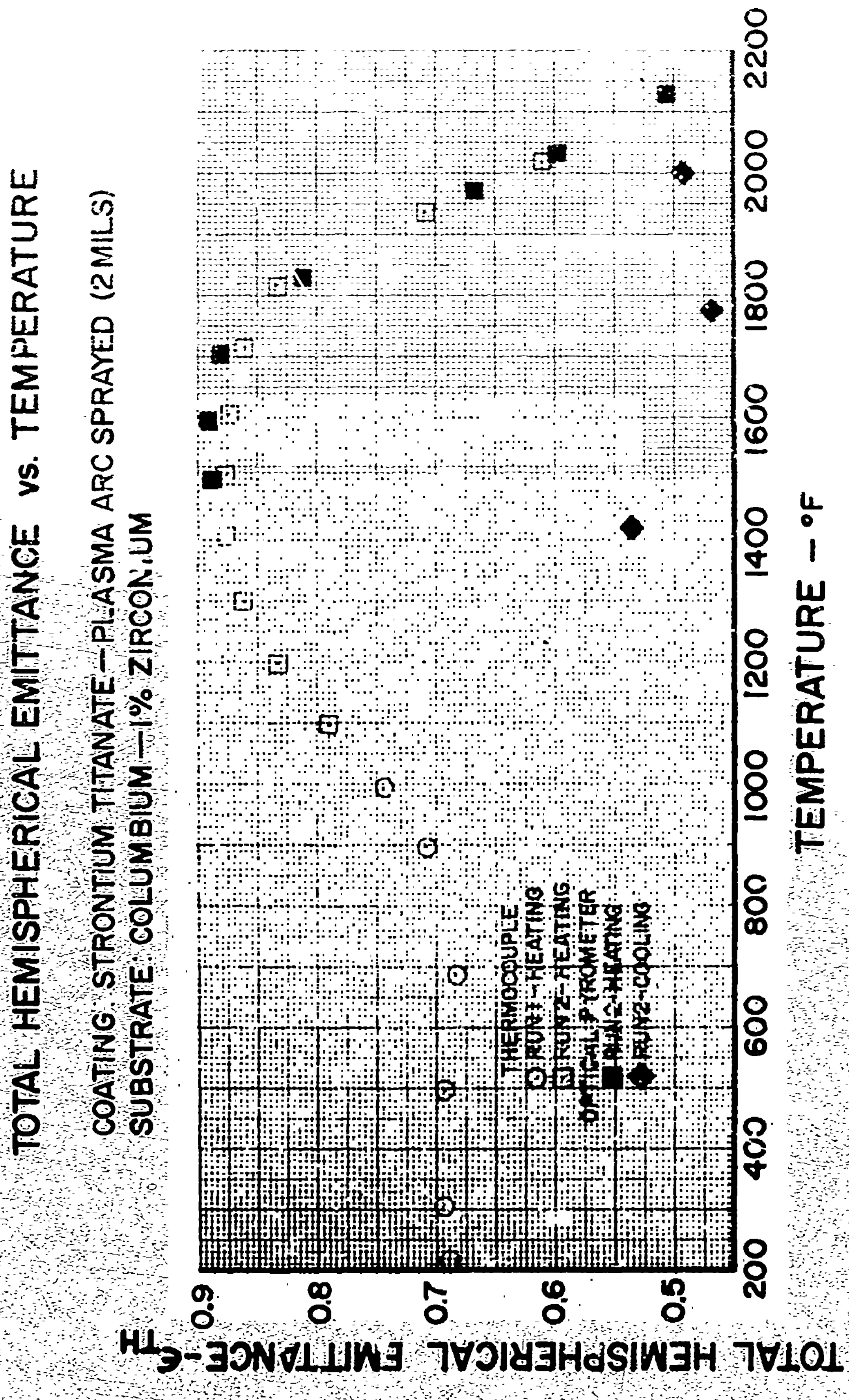

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
COATING: STRONTIUM TITANATE — PLASMA ARC SPRAYED (2 MILS)
SUBSTRATE: COLUMBIUM — 1% ZIRCONIUM
THERMOCOUPLE
RUN 1 - HEATING
RUN 2 - HEATING
OPTICAL PYROMETER
RUN 2 - HEATING
RUN 2 - COOLING
TEMPERATURE — °F
TOTAL HEMISPHERICAL EMITTANCE - ϵ_{TH}
0.9
0.8
0.7
0.6
0.5
200
400
600
800
1000
1200
1400
1600
1800
2000
2200

Figure 30

remained constant. The temperature was changing rapidly enough so that during the time required for obtaining current and voltage readings the temperature changed by several degrees. Thus, some of the discrepancies in the emittance data based on these two types of temperature readings is due to transient conditions.

At temperatures of 2000 °F and higher the platinum-platinum 10 per cent rhodium thermocouple no longer gave reliable data. Therefore only total emittance based on optical pyrometer temperatures are shown for the cooling portion of the test. This data indicates that the decrease in emittance is permanent and exists over the entire temperature range.

Except for a change in color (from light grey to golden brown) no differences in the appearance of the coating before and after testing were noted.

b. Second Specimen - The second coating tested was 4 mils thick, white in color, hard, had a medium grit texture, and was well bonded to the substrate. The specimen was tested over the temperature range of 300 to 2250 °F. The shape of the total emittance curve of this specimen was very similar to that of the first specimen, although the level at certain temperatures was different. This coating had a relatively constant emittance level of 0.74 from 300 to 700 °F. The emittance then increased to a maximum of about 0.9 at 1400 °F and remained relatively constant to 1700 °F. As the specimen temperature was increased above 1700 °F, the emittance decreased until a value of about 0.64 was reached at 2250 °F. This specimen, like the first specimen, and the one reported in PWA-2043, had a much lower emittance as the specimen was cooled. The results of this test are shown in Figure 31.

When the specimen was heated above 2000 °F, the same problem of unreliable temperature measurement with platinum-platinum 10 per cent rhodium thermocouple was encountered. The emittance data based on thermocouple temperatures is included for the final portion of the test. However the emittance values obtained after the data point taken at 2000 °F should be considered to be qualitative and are included only

TABLE XVII

Coating: Strontium Titanate - Plasma Arc Sprayed
Substrate: Columbium - 1% Zirconium

4.0 Mil Coating

Run No.	Elapsed Time (Hrs)	Pressure (mm Hg)	T.C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	0.8	1.9×10^{-7}	300	.732		
	1.1	2.0×10^{-7}	500	.747		
	1.6	2.3×10^{-7}	702	.738		
	1.8	2.4×10^{-7}	901	.755		
	2.8	3.0×10^{-7}	1000	.778		
	3.2	3.1×10^{-7}	1099	.813		
	3.4	2.7×10^{-7}	1199	.853		
	3.6	3.6×10^{-7}	1299	.864		
	3.8	4.0×10^{-7}	1415	.907		
	4.1	3.4×10^{-7}	1497	.894		
	4.3	3.5×10^{-7}	1600	.887	1600	.887
	4.4	3.9×10^{-7}	1700	.884	1710	.884
	4.6	4.6×10^{-7}	1802	.866	1818	.842
	4.7	7.5×10^{-7}	1903	.847	1908	.840
	4.9	9.5×10^{-7}	2005	.798	2007	.796
	5.1	1.4×10^{-6}	2109	.758	2132	.731
	5.2	1.7×10^{-6}	2220	.669	2246	.644
	5.6	1.1×10^{-6}	2154	.613	2190	.581
	6.2	5.0×10^{-7}	1851	.602	1910	.557
	6.6	3.4×10^{-7}	1552	.588	1580	.556
	6.9	2.5×10^{-7}	1250	.569		
	7.2	2.3×10^{-7}	917	.560		
	7.5	2.4×10^{-7}	648	.549		

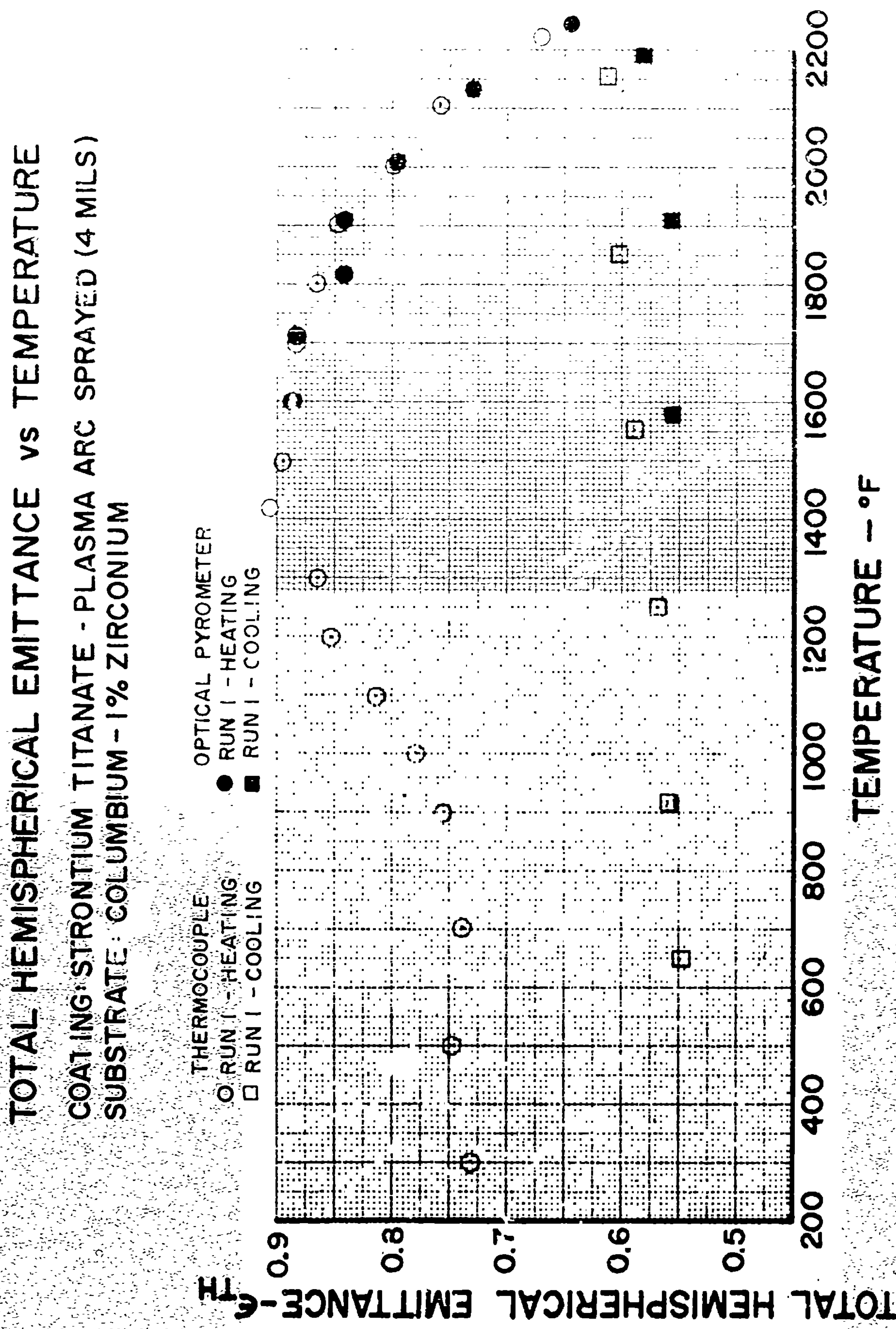

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: STRONTIUM TITANATE - PLASMA ARC SPRAYED (4 MILS)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
RUN I - HEATING
RUN I - COOLING
OPTICAL PYROMETER
RUN I - HEATING
RUN I - COOLING
TOTAL HEMISPHERICAL EMITTANCE - ϵ_{TH}
0.9
0.8
0.7
0.6
0.5
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE — °F

Figure 31

for the purpose of indicating the approximate emittance level of the coating after exposure to higher temperatures. When the specimen was removed from the test rig it was noted that the color of the coating had changed from white to yellow-brown. No other visible characteristics had changed.

Comparing the data for these two specimens shows that the emittance of the second specimen is about 5 per cent higher than that of the first over the temperature range of 300 to 1000°F. From 1100 to 1800°F, the two sets of data agree very well. Above 1800°F the curves start to diverge which is probably a result of the nonrepeatability of the rates of deterioration of the two coatings.

2. Spectral Normal Emittance Test

The specimen tested in the spectral emittance rig was from the same set as the second strontium titanate specimen tested in the total emittance rig. This coating was 4 mils thick, light grey, hard, had a smooth texture and was well bonded to the substrate. A different test sequence was used for this specimen than that used for most specimens tested in the spectral emittance rig. The spectral normal emittance was measured at 900°F from 1.5 to 12.5 microns and at 1450°F from 1 to 13.5 microns. The specimen was then maintained overnight at 1450°F and the spectral emittance was measured again at this temperature over the same wavelength range. The spectral emittance test at 900°F was repeated and the temperature was then raised to 2000°F and spectral emittance was measured from 0.45 to 13.5 microns. As may be seen from Figure 32 there is very little difference in the shapes of the curves. The only difference is that the curves for the second 900°F and 1450°F tests are flatter than for the first tests, thus indicating a possible change in the coating during the overnight endurance period. When the specimen was removed from the rig the only noticeable change was that the color had changed from light grey to light brown.

A spectrographic analysis of the second specimen tested in the total emittance rig and an untested specimen from the same set showed no change in the elements present as a result of testing. X-ray diffraction patterns on these same specimens and the other specimens tested in the total emittance rig show strontium titanate to be the primary phase present in all of the coatings.

SPECTRAL NORMAL EMITTANCE vs WAVELENGTH

COATING: STRONTIUM TITANATE - PLASMA ARC SPRAYED (4 MILS)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM

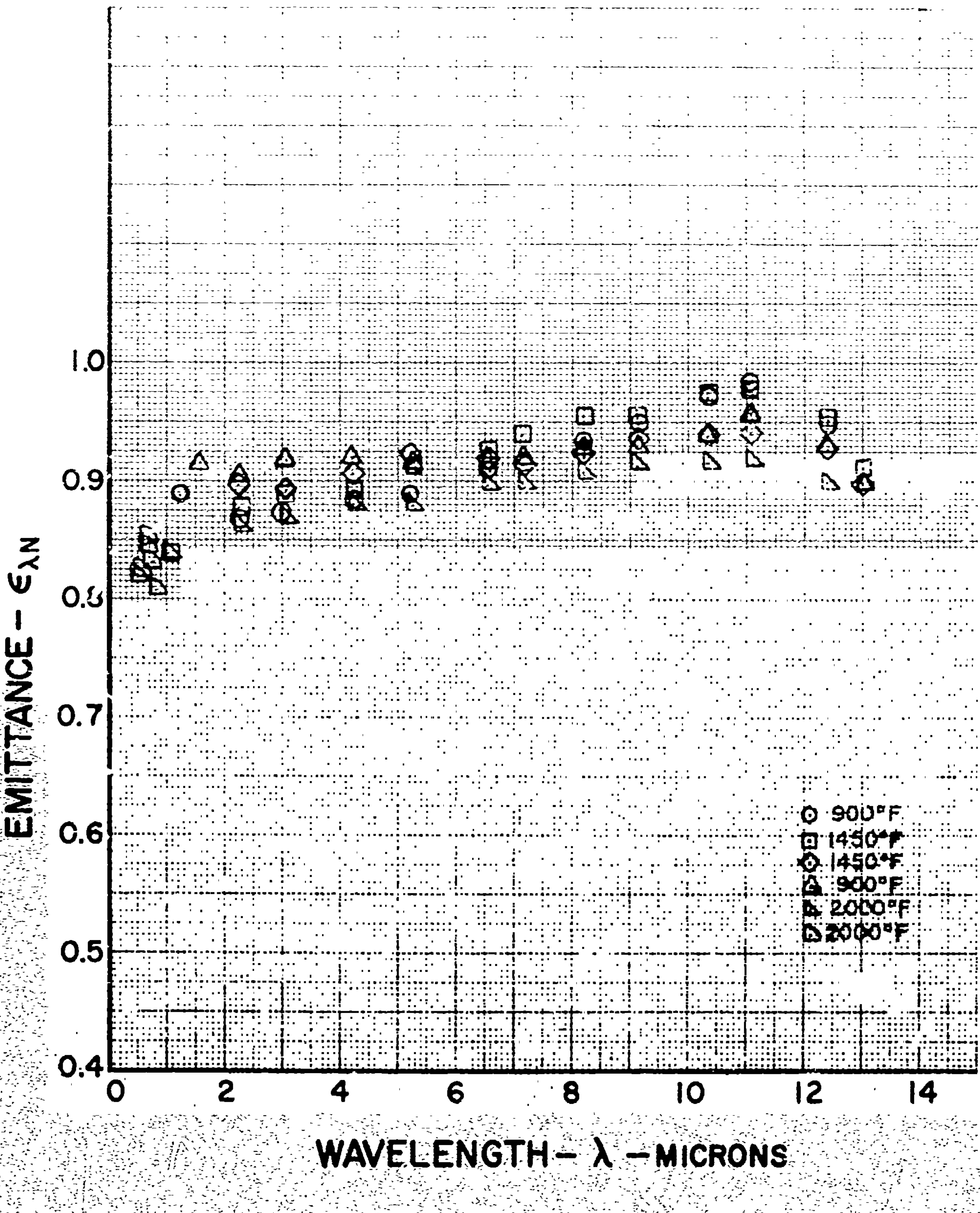


Figure 32

J. Iron Titanium Aluminum Oxide

Powder obtained from the Continental Coatings Corporation (FCT-12) was plasma-arc sprayed onto columbium-1 per cent zirconium tubes for testing in the total hemispherical and spectral normal emittance rigs. X-ray diffraction and spectrographic analyses are being conducted on these coatings.

1. Total Hemispherical Emittance Test

The 5-mil thick coating was light black, fairly hard, had a medium grit texture, and a poor substrate bond. The total emittance of the coating varied from about 0.83 at 300°F to about 0.88 at 900°F and then remained relatively constant up to 2150°F. The emittance values obtained during cooling were about the same as those obtained during heating. The data are shown in Figure 33.

Optical pyrometer readings were unreliable throughout the test and therefore measurements of temperatures by the platinum-platinum 10 per cent rhodium thermocouple are the only ones reported. It is believed that no thermocouple contamination occurred. Had contamination occurred, it would have been accelerated at the higher temperatures and would have resulted in measured emittance values during cooling which differed from those measured during heating. When the specimen was removed from the rig the only noticeable change was that the color had slightly darkened.

2. Spectral Normal Emittance Test

The 4-mil thick coating was dark grey, fairly hard, had a medium grit texture, and was poorly bonded to the substrate. The testing procedure used for this specimen was the same as that used for strontium titanate.

Spectral normal emittance was measured from 1.5 to 12.5 microns at 900°F and from 1.0 to 13.5 microns at 1450°F. The specimen was then maintained overnight at 1450°F and spectral emittance was measured again at these temperatures. Finally, the test temperature was raised to 2000°F and spectral emittance was measured from 0.45 to 13.5 microns.

The results of this test are presented in Figure 34. There was no change in the external characteristics of the specimen as a result of testing.

TABLE XVIII

Coating: Iron-Titanium-Aluminum-Oxide
Plasma-Arc Sprayed
Substrate: Columbium-1% Zirconium

5.0 Mil Coating

Run No.	Elapsed Time (Hrs.)	Pressure (mm Hg)	T.C. (°F)	ϵ_{TH}
1	2.0	7.7×10^{-7}	299	.831
	2.7	1.3×10^{-6}	497	.855
	3.0	1.8×10^{-6}	699	.864
	4.0	2.3×10^{-6}	903	.879
	4.5	4.0×10^{-6}	998	.876
	4.8	8.0×10^{-6}	1100	.874
	5.1	5.3×10^{-6}	1198	.880
	5.3	5.0×10^{-6}	1300	.878
	5.6	5.6×10^{-6}	1400	.872
	6.0	1.5×10^{-5}	1499	.879
	6.4	8.0×10^{-6}	1599	.875
	6.7	8.0×10^{-6}	1699	.872
	7.0	3.1×10^{-5}	1799	.865
	7.3	2.8×10^{-5}	1897	.871
	7.8	4.4×10^{-5}	1999	.882
	8.0	1.5×10^{-5}	2097	.888
	8.3	1.1×10^{-5}	2148	.891
	8.5	9.0×10^{-6}	1850	.901
	8.7	4.2×10^{-6}	1551	.893
	8.8	3.1×10^{-6}	1251	.881
	9.1	5.0×10^{-6}	928	.867

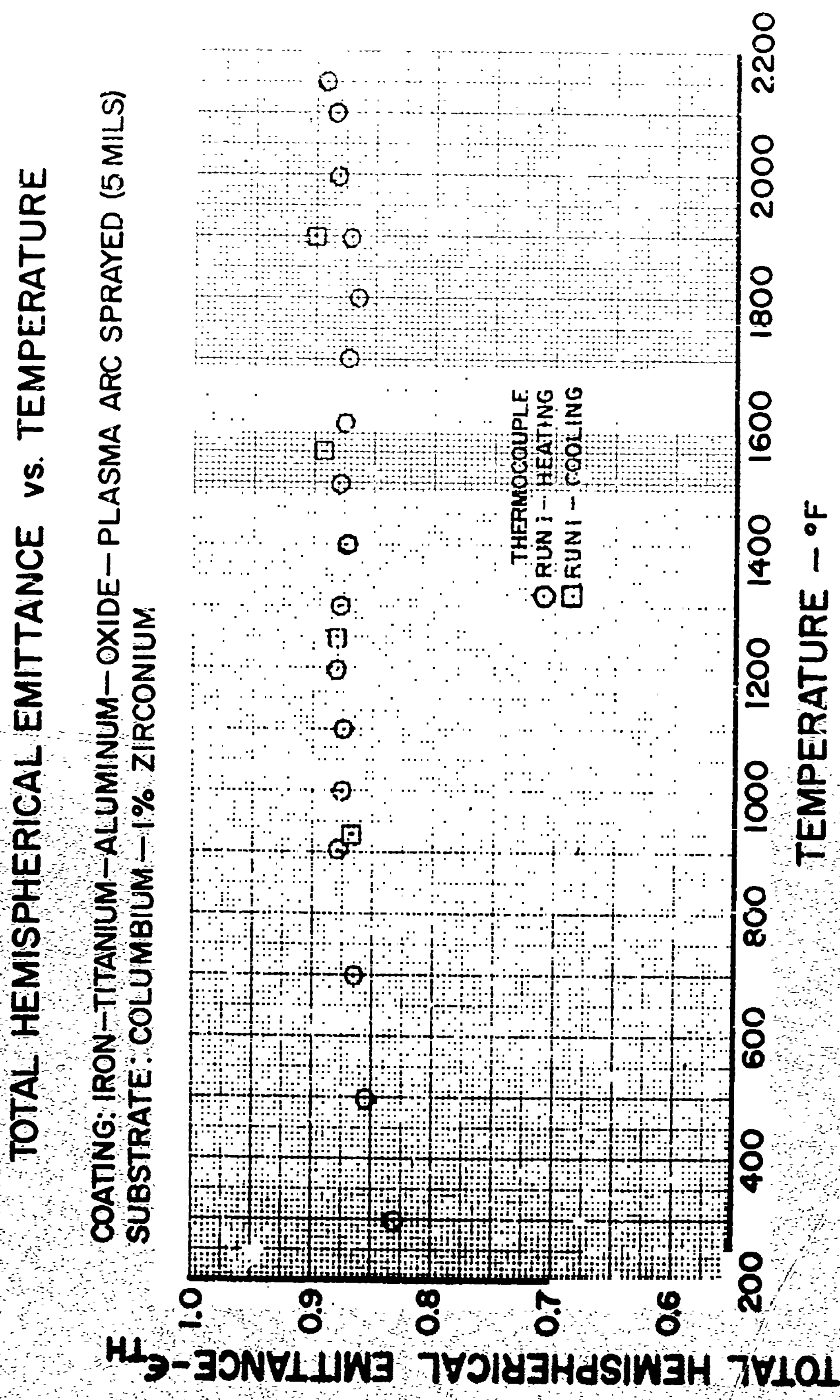

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
COATING: IRON-TITANIUM-ALUMINUM-OXIDE-PLASMA ARC SPRAYED (5 MILS)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM
THERMOCOUPLE
RUN I - HEATING
RUN I - COOLING
TOTAL HEMISPHERICAL EMITTANCE-ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE — °F

Figure 33

SPECTRAL NORMAL EMITTANCE vs WAVELENGTH

COATING: IRON-TITANIUM-ALUMINUM-OXIDE-PLASMA ARC SPRAYED (4 MILS)
SUBSTRATE: COLUMBIUM-1 % ZIRCONIUM

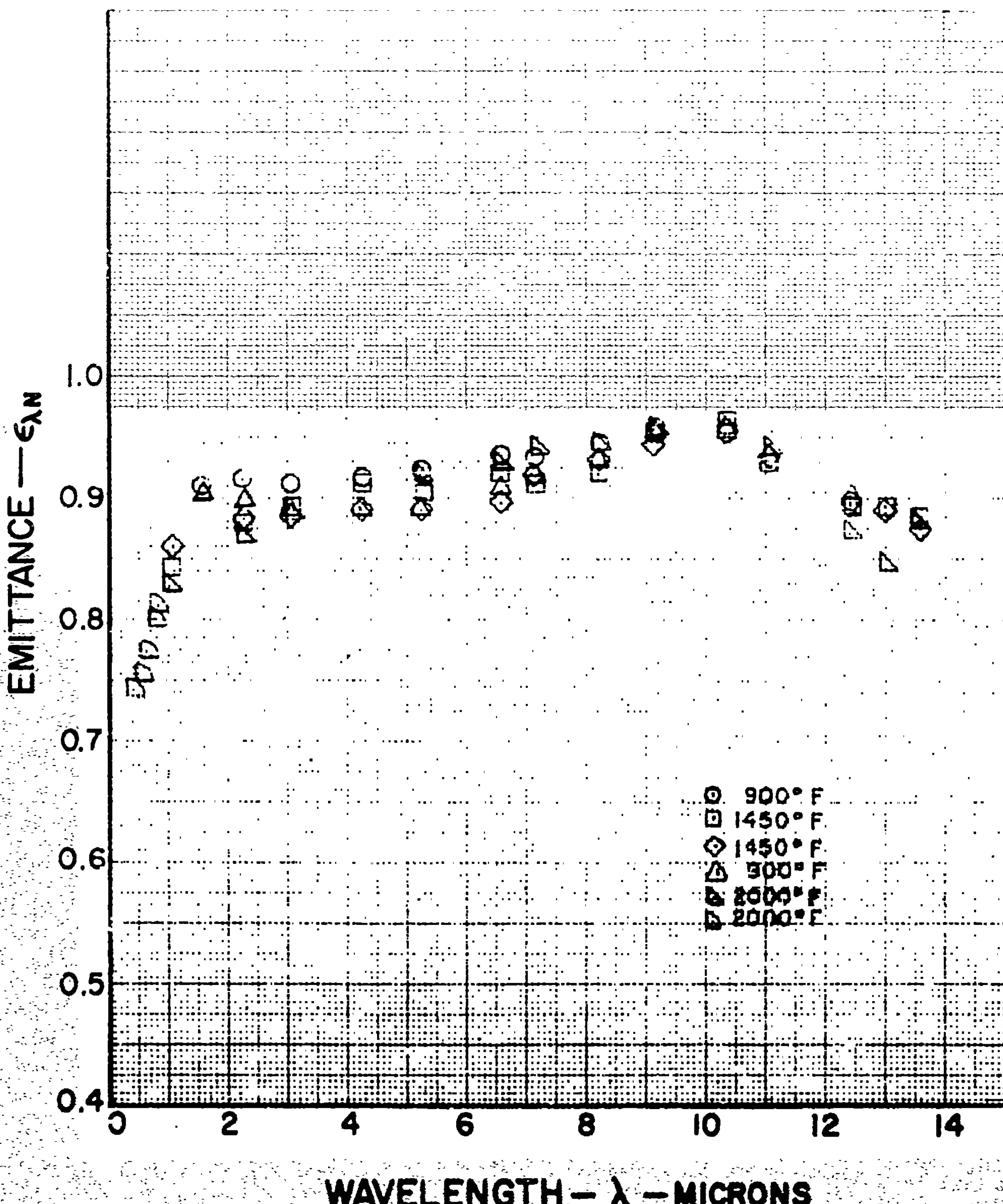


Figure 34

K. Iron-Titanium Oxide

Powder obtained from Continental Coatings Corporation (FCT-11) was plasma-arc sprayed onto columbium-1 percent zirconium tubes. The specimens were tested in the total hemispherical emittance rig and in the short term endurance rig.

1. Total Hemispherical Emittance Test

The 4-mil thick coating was grey-black and hard, and had a medium grit texture and a good substrate bond. Total hemispherical emittance was measured over the temperature range of 300 to 2100°F and was relatively constant at approximately 0.85 over the entire temperature range. Several test runs were made with no appreciable changes in emittance resulting from repeated heating. (See Figure 35). Optical pyrometer readings during runs 2 and 3 were found to be unreliable and the data based on these temperatures are not reported.

2. Short Term Endurance Test

The coating tested in the short term endurance rig was 4 mils thick, grey-black, hard, had a medium matte grit texture and a good substrate bond. Figure 36 shows the total hemispherical emittance of the specimen as it was heated from 300°F to 1450°F. The specimen was subjected to 300 hours of endurance testing at 1450°F. The results of the endurance testing are shown in Figure 37.

These two figures show that the emittance of the sample was relatively constant at 0.87 from 800 to 1450°F and that no significant change occurred during the 300 hours at 1450°F.

No change in the coating was observed as a result of testing. X-ray diffraction and spectrographic analyses are being conducted on the specimens.

TABLE XIX

Coating: Iron Titanium Oxide Plasma-Arc Sprayed
Substrate: Columbium-1 Per Cent Zirconium

4.0 Mil Coating

Run No.	Elapsed Time (Hrs.)	Pressure (mm Hg)	T.C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	1.5	1.6×10^{-7}	300	.822		
	4.0	1.6×10^{-7}	505	.856		
	5.0	1.6×10^{-7}	695	.836		
	5.6	2.6×10^{-7}	904	.854		
	6.1	3.2×10^{-7}	1003	.863		
Heating Current Off; Vacuum Maintained						
2	6.8	3.6×10^{-7}	1000	.881		
	7.2	3.5×10^{-7}	1099	.871		
	7.7	2.6×10^{-7}	1200	.873		
	8.2	2.2×10^{-7}	1300	.874		
	8.7	2.0×10^{-7}	1400	.882		
	10.6	1.1×10^{-6}	1500	.875		
	10.9	1.0×10^{-6}	1596	.867		
	11.3	2.2×10^{-6}	1699	.865		
	11.6	3.3×10^{-6}	1799	.893		
	12.3	3.4×10^{-6}	1902	.914		
Heating Current Off; Vacuum Chamber Opened						
3	13.6	1.2×10^{-6}	1001	.868		
	14.0	2.6×10^{-6}	1494	.843		

TABLE XIX (CONTINUED)

Run No.	Elapsed Time (Hrs.)	Pressure (mm Hg)	T. C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
Heating Current Off; Vacuum Chamber Opened; Flange Removed For Repairs						
4	15.4	2.4×10^{-7}	1500	.860	1492	.874
	15.5	2.6×10^{-7}	1599	.848	1600	.846
	15.6	3.0×10^{-7}	1700	.846	1701	.847
	15.7	3.2×10^{-7}	1799	.845	1790	.857
	15.7	4.8×10^{-7}	1899	.845	1910	.829
	15.8	6.0×10^{-7}	2001	.849	2008	.839
	15.9	2.0×10^{-6}	2095	.879	2088	.888
	16.2	4.9×10^{-7}	1901	.858	1900	.359
	16.3	2.8×10^{-7}	1701	.866	1709	.869
	16.4	2.5×10^{-7}	1500	.866	1508	.849

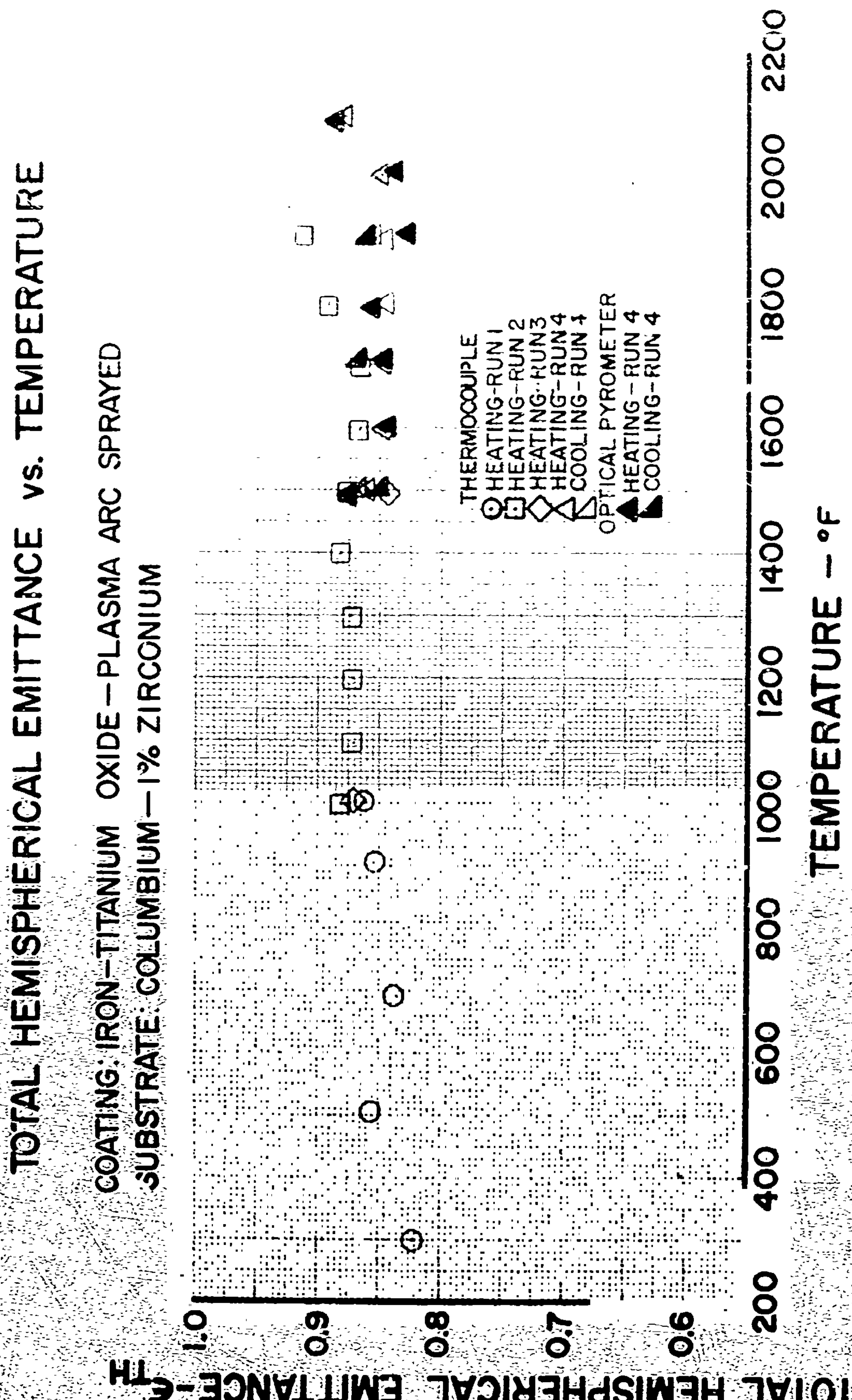

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
COATING: IRON-TITANIUM OXIDE—PLASMA ARC SPRAYED
SUBSTRATE: COLUMBIUM—1% ZIRCONIUM
THERMOCOUPLE
HEATING-RUN 1
HEATING-RUN 2
HEATING-RUN 3
HEATING-RUN 4
COOLING-RUN 4
OPTICAL PYROMETER
HEATING-RUN 4
COOLING-RUN 4
TOTAL HEMISPHERICAL EMITTANCE-ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE — °F

Figure 35

TABLE XX

Coating: Iron Titanium Oxide-Plasma Arc Sprayed
Substrate: Columbium-1 Per Cent Zirconium

4 Mil Coating

Run No.	Elapsed Time (Hrs.)	Pressure (mm Hg)	T.C. (°F)	ϵ_{TH}	Optical (°F)	ϵ_{TH}
1	4.0	4.6×10^{-8}	297	.895		
	4.2	5.0×10^{-7}	399	.895		
	4.5	1.1×10^{-6}	499	.895		
	4.7	1.7×10^{-6}	597	.881		
	5.0	2.1×10^{-6}	698	.881		
	5.2	1.9×10^{-6}	800	.866		
	5.4	3.5×10^{-6}	898	.875		
	5.7	3.9×10^{-6}	1001	.865		
	5.9	3.4×10^{-6}	1100	.869		
	6.2	2.9×10^{-6}	1199	.869		
	6.8	5.7×10^{-6}	1398	.862		
	7.1	5.7×10^{-6}	1454	.857	1440	.883

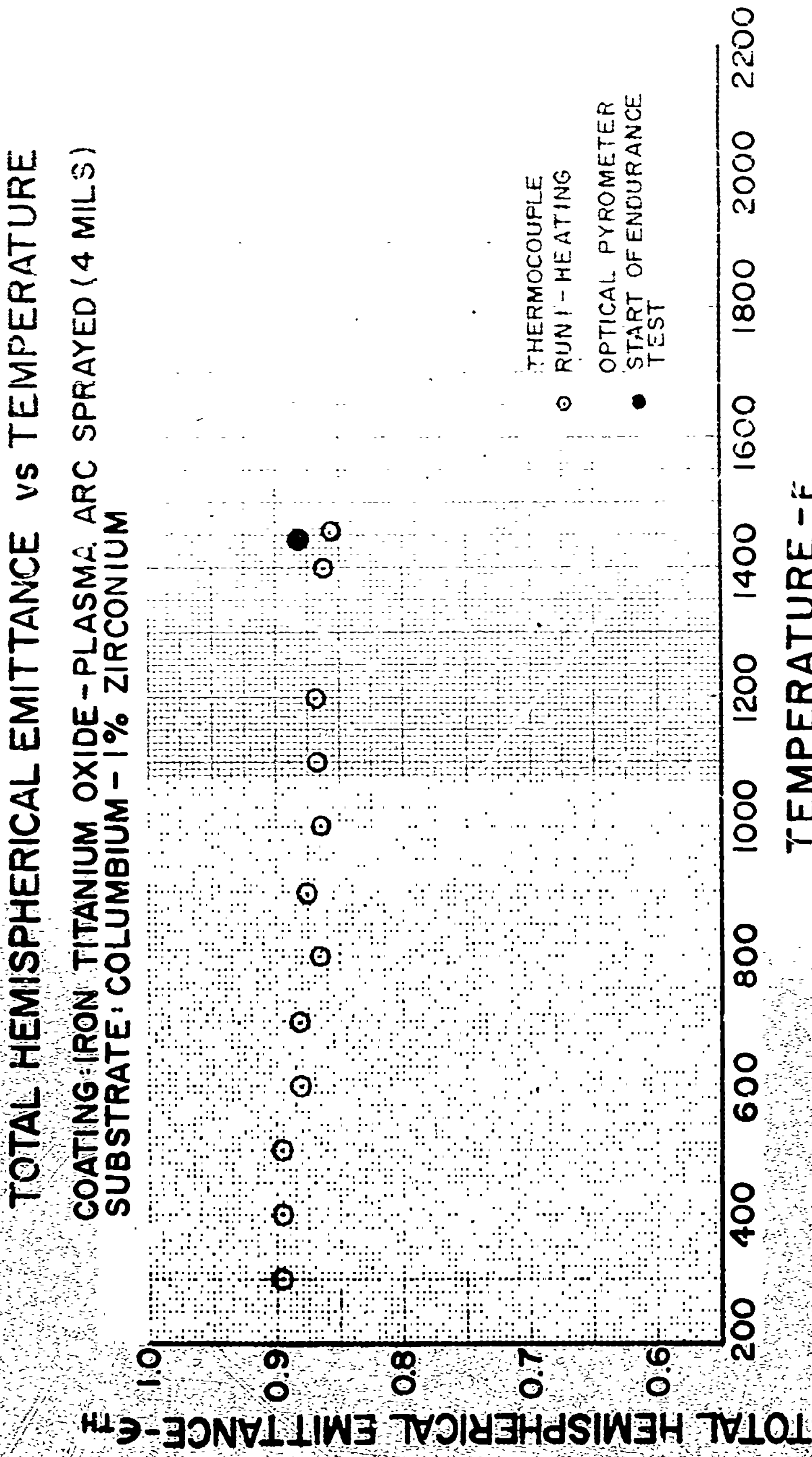

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: IRON TITANIUM OXIDE - PLASMA ARC SPRAYED (4 MILS)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
RUN I - HEATING
OPTICAL PYROMETER
START OF ENDURANCE TEST
TOTAL HEMISPHERICAL EMITTANCE - ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE - F

Figure 36

TABLE XXI

Coating: Iron Titanium Oxide-Plasma Arc Sprayed
Substrate: Columbium-1 Per Cent Zirconium

4.0 Mil Coating

Run No.	Elapsed Time (Hrs.)	Pressure (mm Hg)	T. C. (°F)	ϵ_{TH}
1	2.5	5.7×10^{-6}	1440	.883
	20.3	1.7×10^{-7}	1439	.883
	40.6	1.0×10^{-7}	1439	.887
	60.99	6.9×10^{-8}	1435	.893
	140.3	4.7×10^{-8}	1439	.897
	160.55	4.3×10^{-8}	1438	.875
	180.8	3.4×10^{-8}	1440	.870
	210.25	3.1×10^{-8}	1440	.867
	230.65	3.1×10^{-8}	1440	.870
	300.5	3.1×10^{-8}	1440	.871

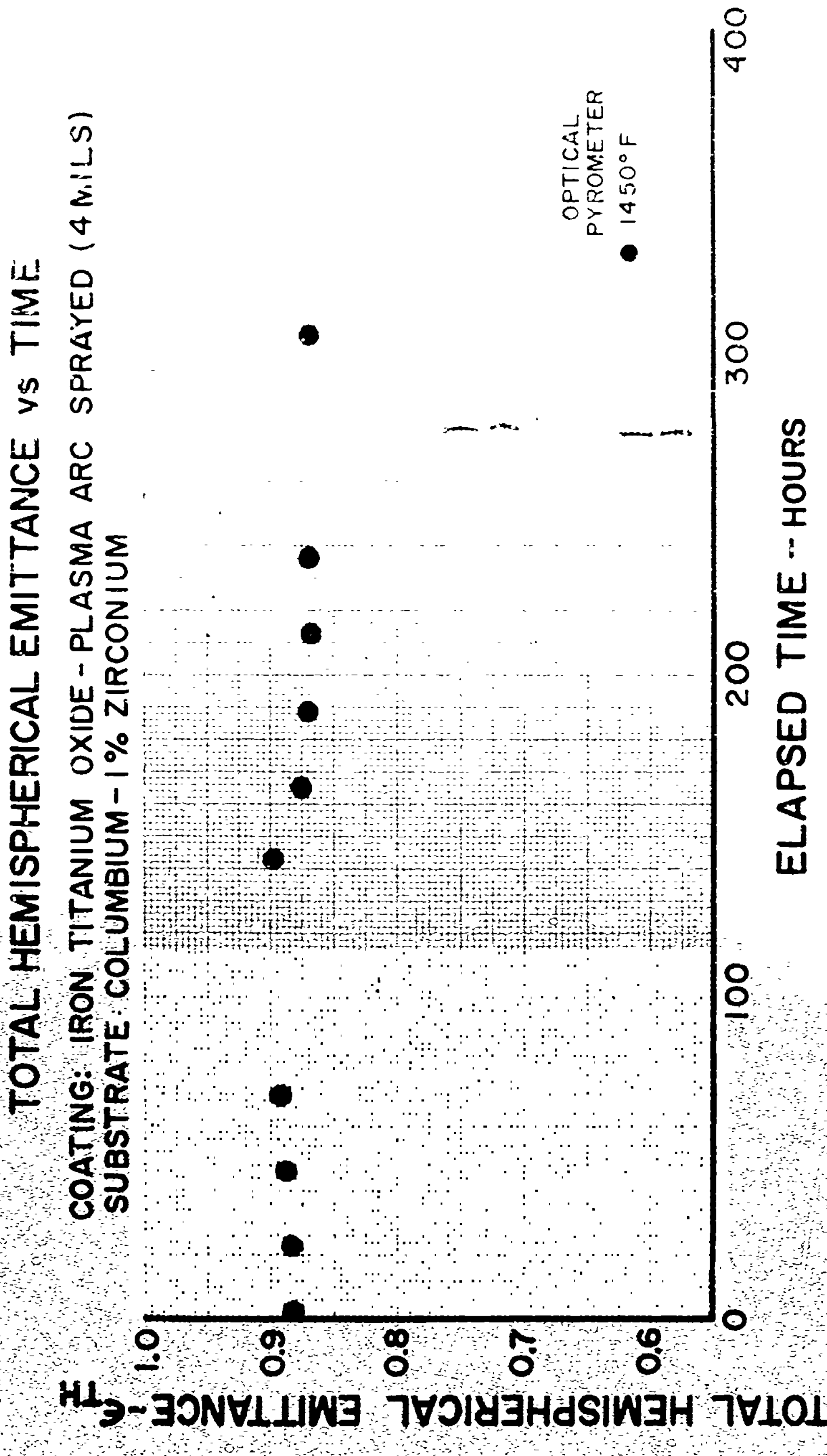

TOTAL HEMISPHERICAL EMITTANCE vs TIME
COATING: IRON TITANIUM OXIDE - PLASMA ARC SPRAYED (4 MILS)
SUBSTRATE COLUMBIUM - 1% ZIRCONIUM
OPTICAL PYROMETER
1450°F
TOTAL HEMISPHERICAL EMITTANCE - ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
0
100
200
300
400
ELAPSED TIME - HOURS

Figure 37